

Purpose

The purpose of this experiment was to characterize Ugi Products 233 and 234 using Fourier Transformed (FT) Nuclear Magnetic Resonance (NMR) Spectroscopy, FT Infrared (IR) Spectroscopy, HyperChem (Version 7.5) Software, and comparison with the results from previously analyzed Ugi 173G.

Experimental

The Jeol 300 MHz FT-NMR was used to determine the purity of the solvents and collect several types of spectrum. The following spectra were collected: H^1 NMR, C^{13} decoupled NMR, DEPT decoupled, Distortionless Enhancement by Polarization Transfer (DEPT) decoupled, Double Quantum Filtered Correlation Spectroscopy (DQF COSY), Heteronuclear shift correlation (chshf), and Nuclear Overhauser Effect Spectroscopy (NOESY). For the FT-IR Spectroscopy, a ThermoNicolet Avatar 360 FT-IR was used with the pressed KBr disc method. Proton and Carbon shifts were predicted using the HyperChem (Version 7.5) and HyperNMR Software on both the predicted rotamers of the two Ugi Products at absolute zero and at room temperature for 0.1 ps. The results were then compared with the Ugi 173G results from the Useful Chemistry Experiment 173, performed by the Drexel University Chemistry Ph-D student Khalid Mirza.

First, the NMR tubes were cleaned and desiccated until the impurities in the spectra became negligible. Then the NMR was auto-gradient shimmed and locked, followed by the tuning and matching of both the proton and carbon probes. Ugi 233 and Ugi 234 were then analyzed in chloroform-d by H^1 NMR, C^{13} decoupled NMR, DEPT decoupled, DEPT decoupled, DQF COSY, Heteronuclear shift correlation, and NOESY. The structures of the two Ugi products were then drawn and labeled using HyperChem. All atoms were selected to be quantum atoms

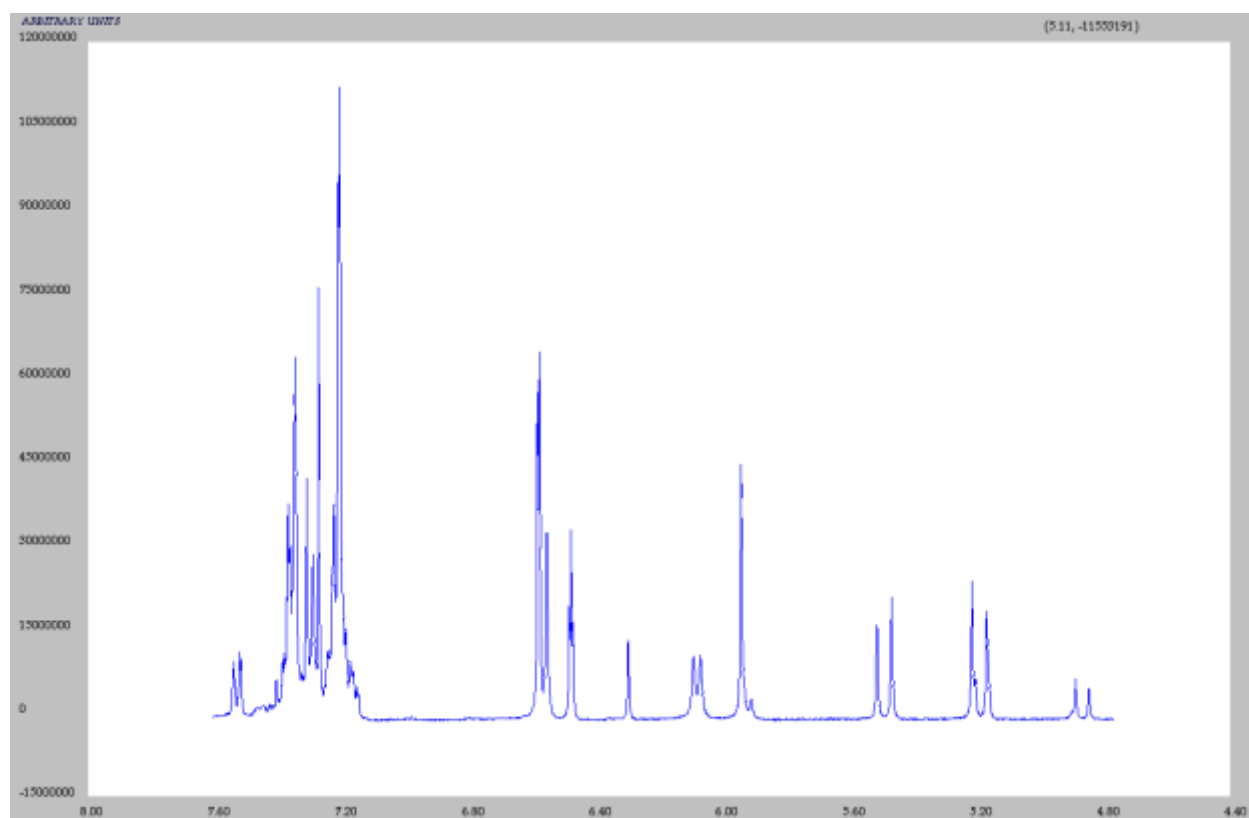
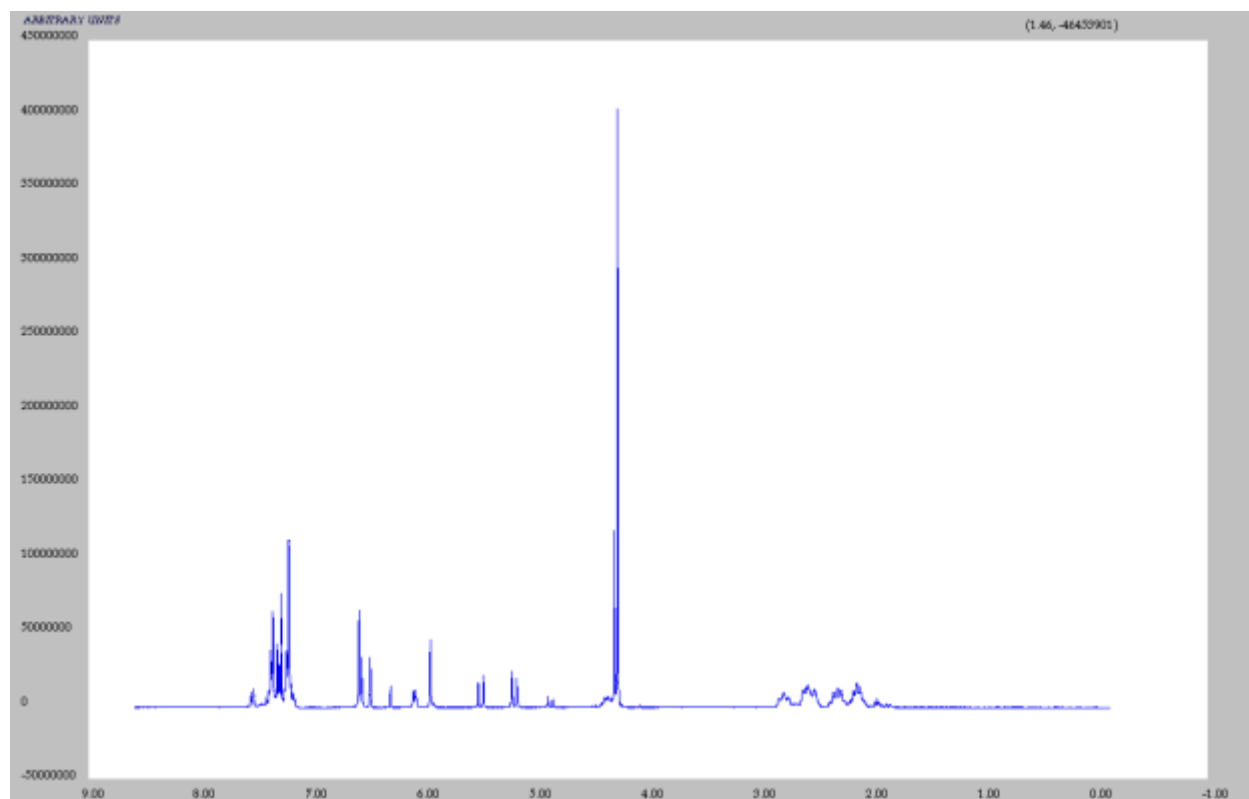
and only the protons were selected to be NMR atoms, except for one with all atoms being NMR atoms for Ugi 234. HyperNMR predicted the chemical shifts corresponding to each atom, following the molecular dynamics (0 K, then 0.1 ps of 300 K) of the Ugi Products. Next, solid KBr pellets were made of the two Ugi Products' remaining solid using the pressed-disc method and analyzed in the FT-IR. Finally, the FT-IR and NMR spectra were compared with those from Ugi 173G, which has had its structure previously confirmed.

Data and Results

300 MHz shift of Ugi 233	Splitting of Ugi 233	J-J Coupling Constant of Ugi 233	Description of Ugi 233	500 MHz shift of Ugi 173 G	Splitting of Ugi 173G	J-J Coupling Constant of Ugi 173G	Description of Ugi 173G	300 MHz shift of Ugi 234	Splitting of Ugi 234	J-J Coupling Constant of Ugi 234	Description of Ugi 234
0.65-0.98	m	10.2	cyclohexane	0.79	t	7.3	minor (3H)	0.88-1.1	m	10.2	cyclohexane
0.98-1.2	m		cyclohexane	0.88	t	7.3	Major (3H)	1.01-1.25	m/s		cyclohexane
1.2-1.42	m(5)	10.2	cyclohexane	1.13 - 1.23	m	7.3	minor (2H)	1.25-1.45	q (4)	10.2	cyclohexane
1.48-1.75	m		cyclohexane	1.24 - 1.33	m	7.3	Major (2H)	1.5-1.75	m		cyclohexane
1.8-1.98	t	12.3	cyclohexane	1.37 - 1.50	m	7.3	2H	1.8-2.0	m/d	10.2	cyclohexane
				3.12 - 3.24	m	7	1H				
				3.23 - 3.34	m	7	1H				
3.65	s			3.65	s		M (6H)				
3.685	s			3.66	s		m (6H)				
3.7-3.85	m										
4.335-4.4	m							4.29-4.44	m	3.3	
4.73,4.79	d	12.30		4.59	d	15.1	m (1H)	4.911,4.953	d	12.9	
5.09,5.15	d	12.3		4.66	d	15.1	m (1H)	5.259,5.303	d	12.9	
5.167,5.214	d	13.8		4.78	d	16.6	M (1H)	5.313,5.359	d	13.8	
5.467,5.513	d	13.8		5.14	d	16.6	M (1H)	5.566,5.520	d	13.8	
5.91	s			5.68	s		M (1H)	6.085,6.108	d	6.9	
5.94	s			5.97	t	5.8	m (1H)	6.198	s		
6.068,6.091	d	6.9		6.04	t	5.8	M (1H)	6.327	s		

6.395	s			6.12	s		m (1H)				
6.694,6.4755,6.4816	t	1.83		6.29 - 6.34	m		M (1H)				
6.552	s			6.35 - 6.37	m		m (1H)				
6.575,6.581	d	1.83		6.4	d	2.4	m (1H)	6.461,6.484	d	6.9	
				6.43	d	2.4	M (1H)				
				7.09	dd	7.8, 1.4	(1H)	7.028-7.227	m		
7.176-7.257	m			7.14 - 7.23	m		(5H)	7.26	s		Solvent
7.257-7.322	m			7.24 - 7.31	m		(2H)	7.276-7.432	m		
7.322-7.392	m			7.32 - 7.48	m		(2H)	7.447-7.488	m		
7.5167,7.5377	d	6.3		7.56	d	7.8	(H)	7.569	s		
								7.624	s		
								7.654	s		

Table 1: Proton NMR Results of Ugi 233, Ugi 173G, and Ugi 234



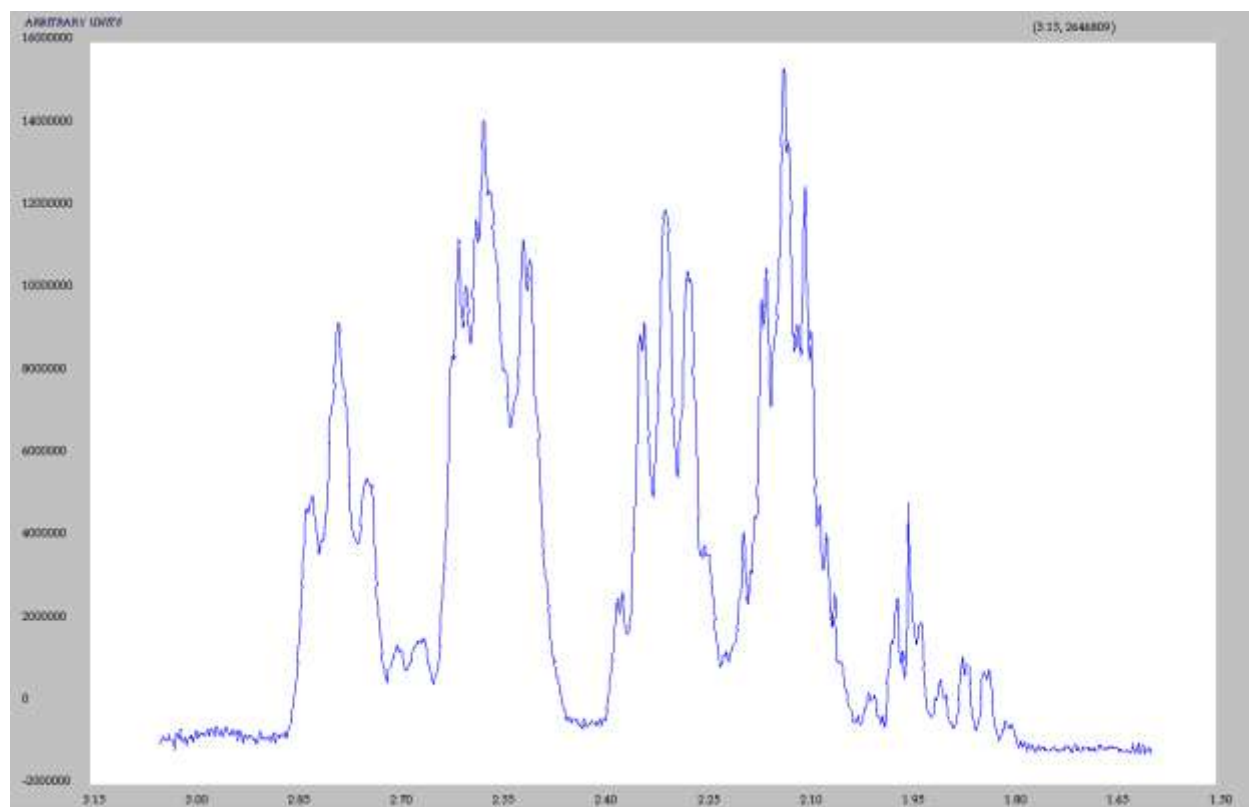
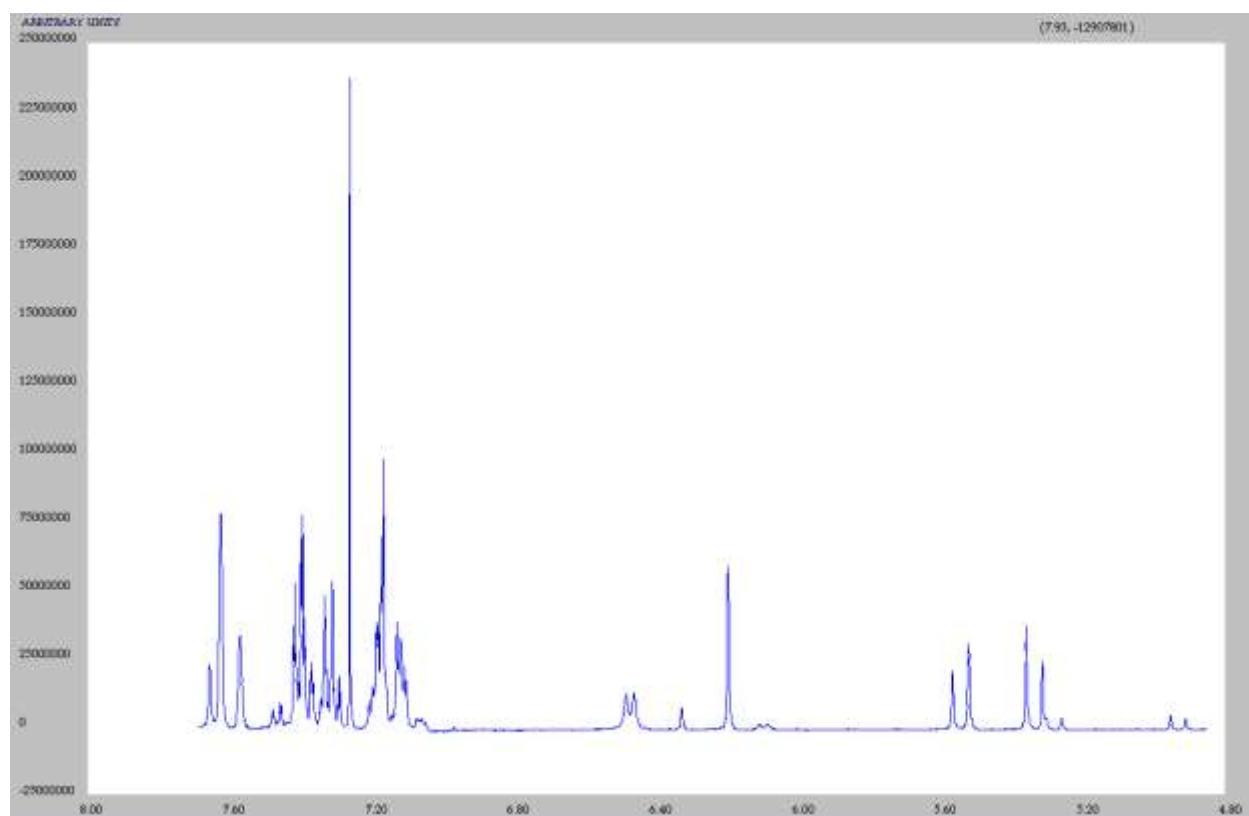
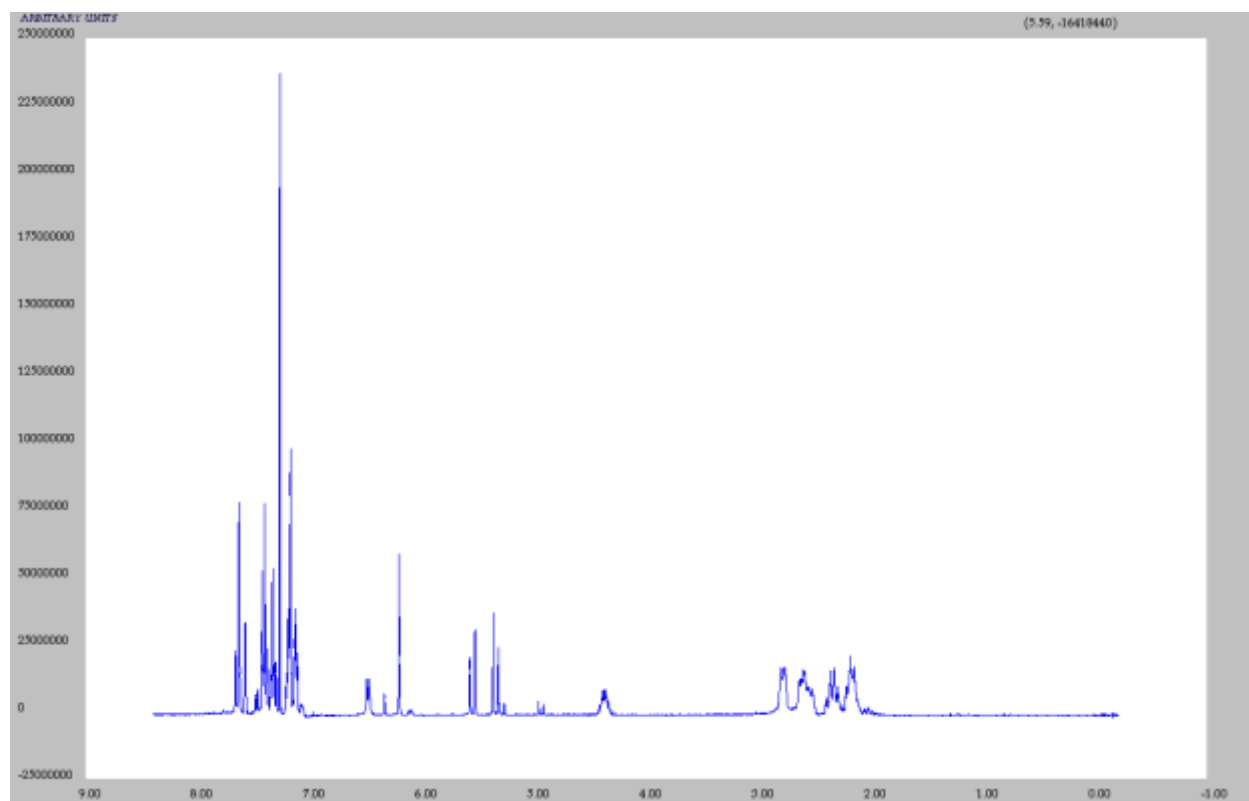


Figure 1: Proton NMR of Ugi 233



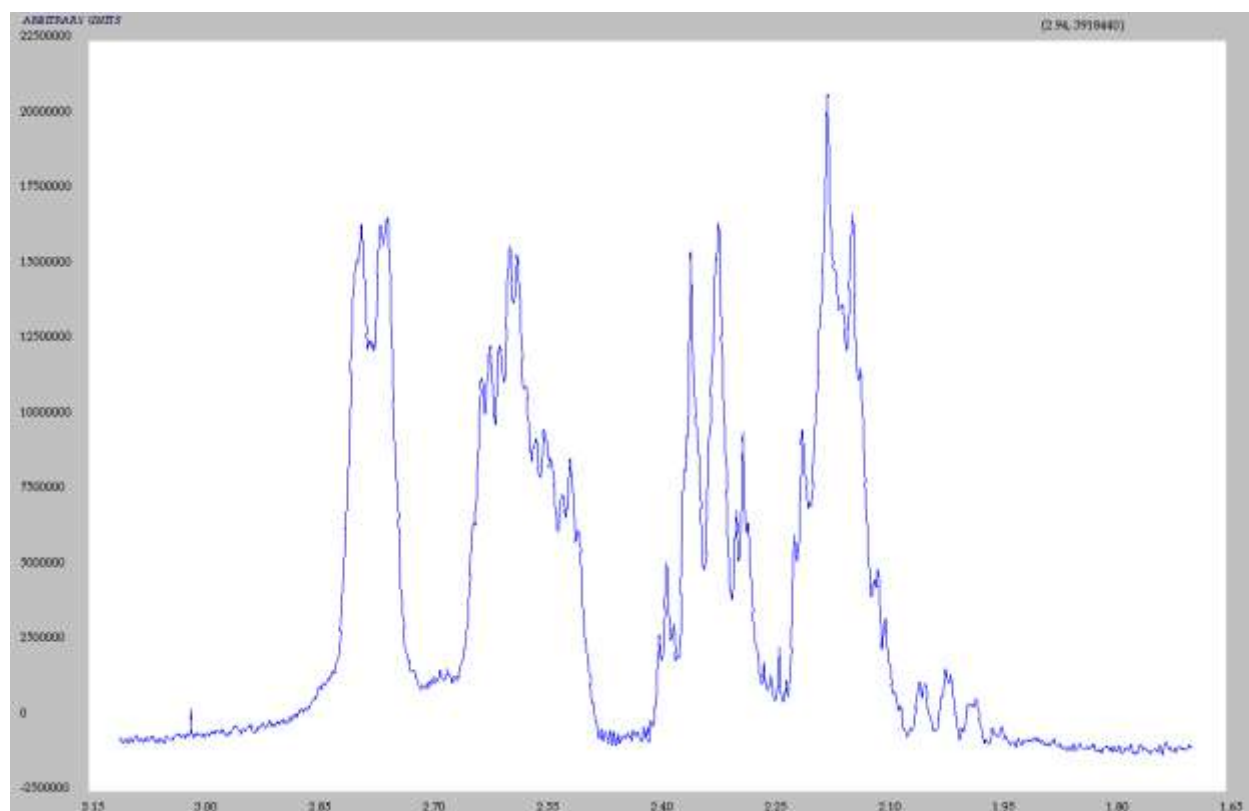
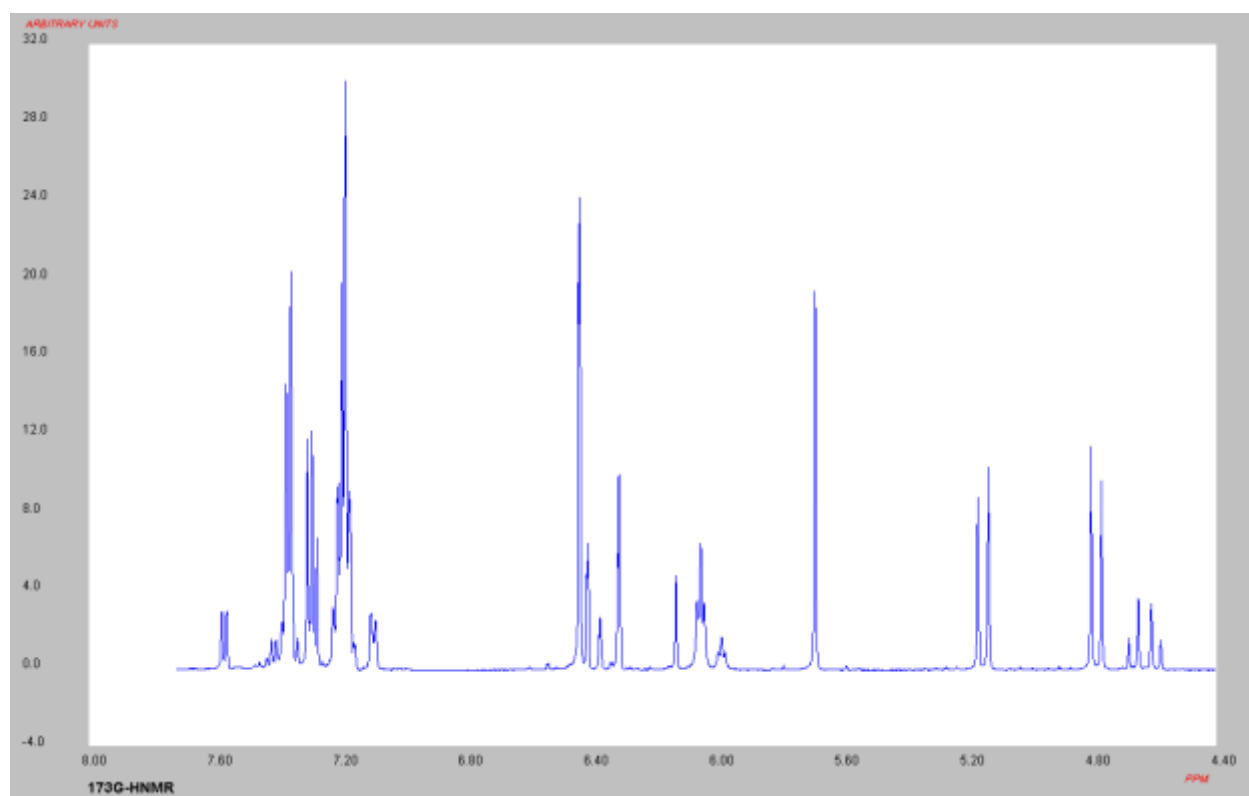
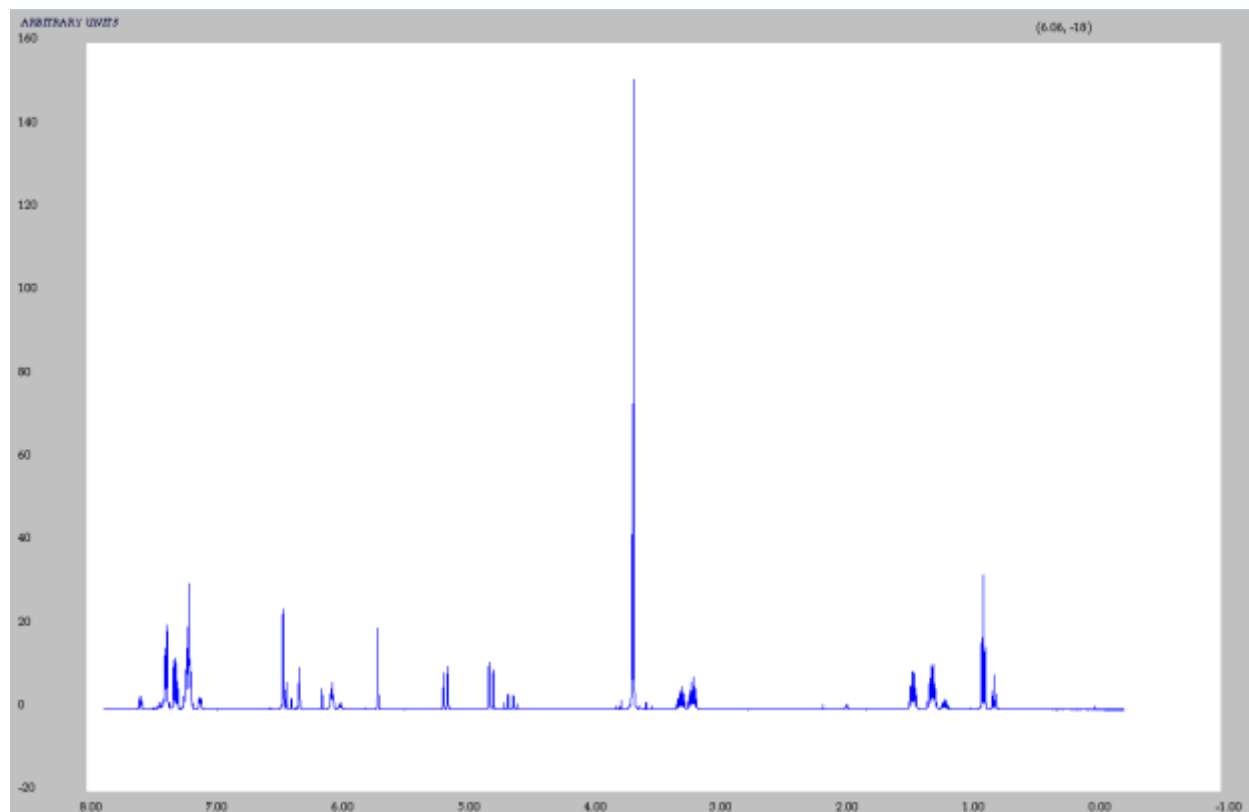


Figure 2: Proton NMR of Ugi 234



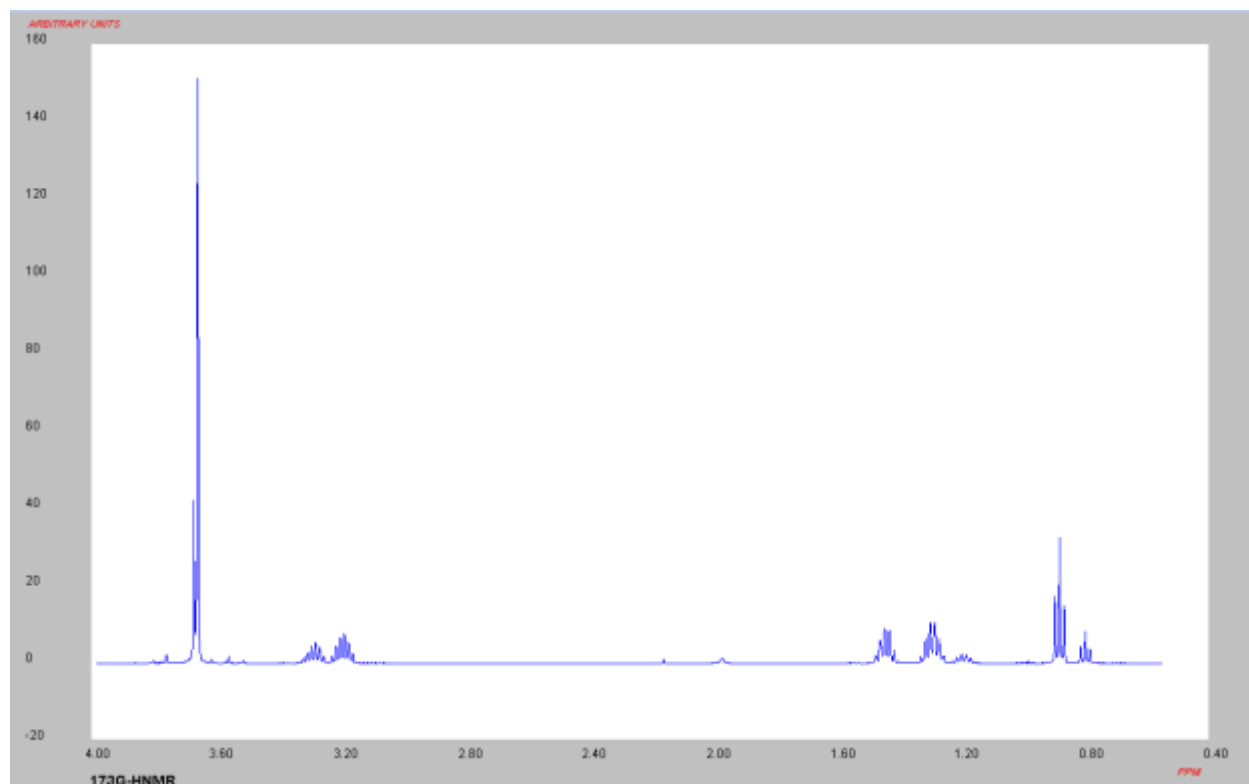


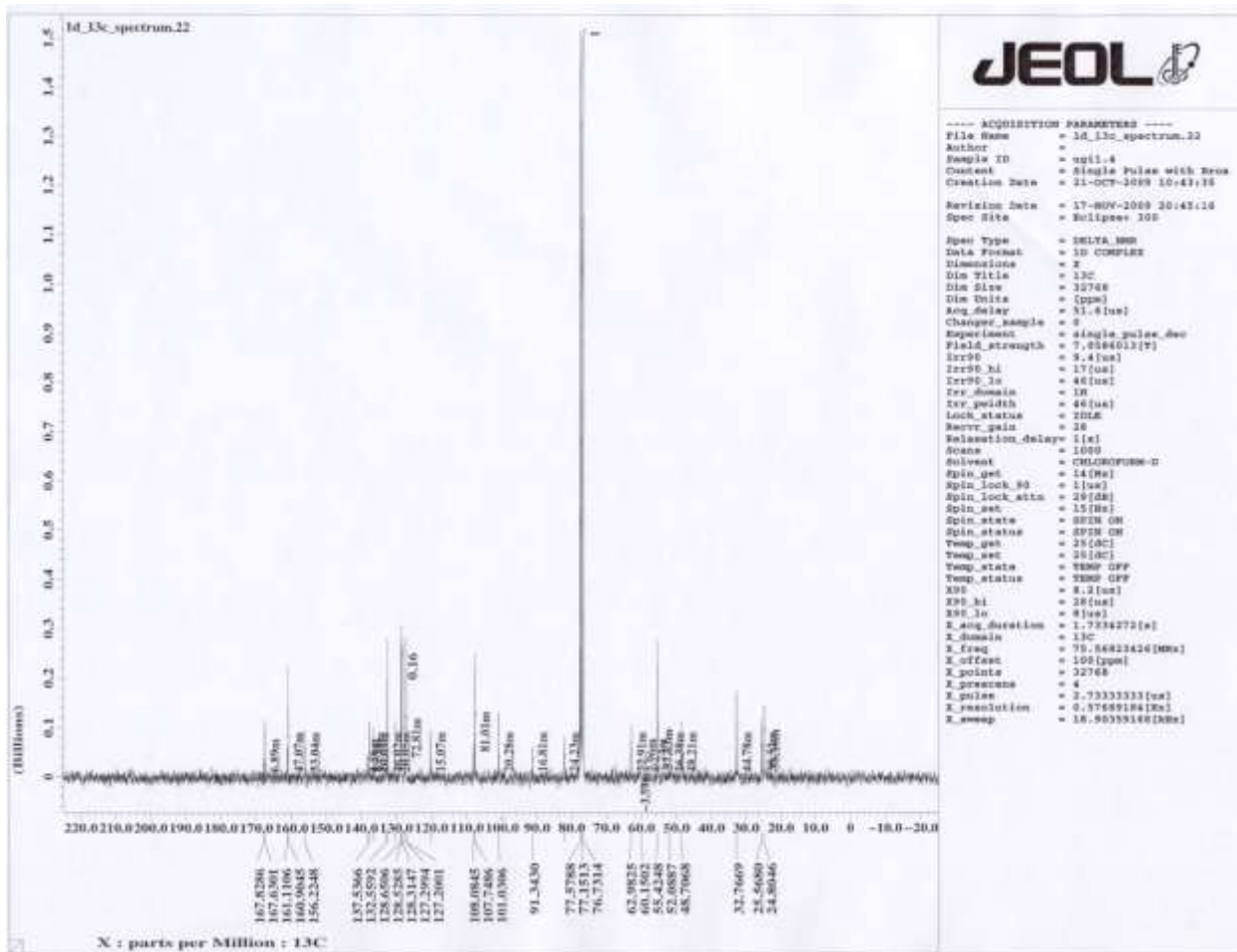
Figure 3: Proton NMR of Ugi 173G

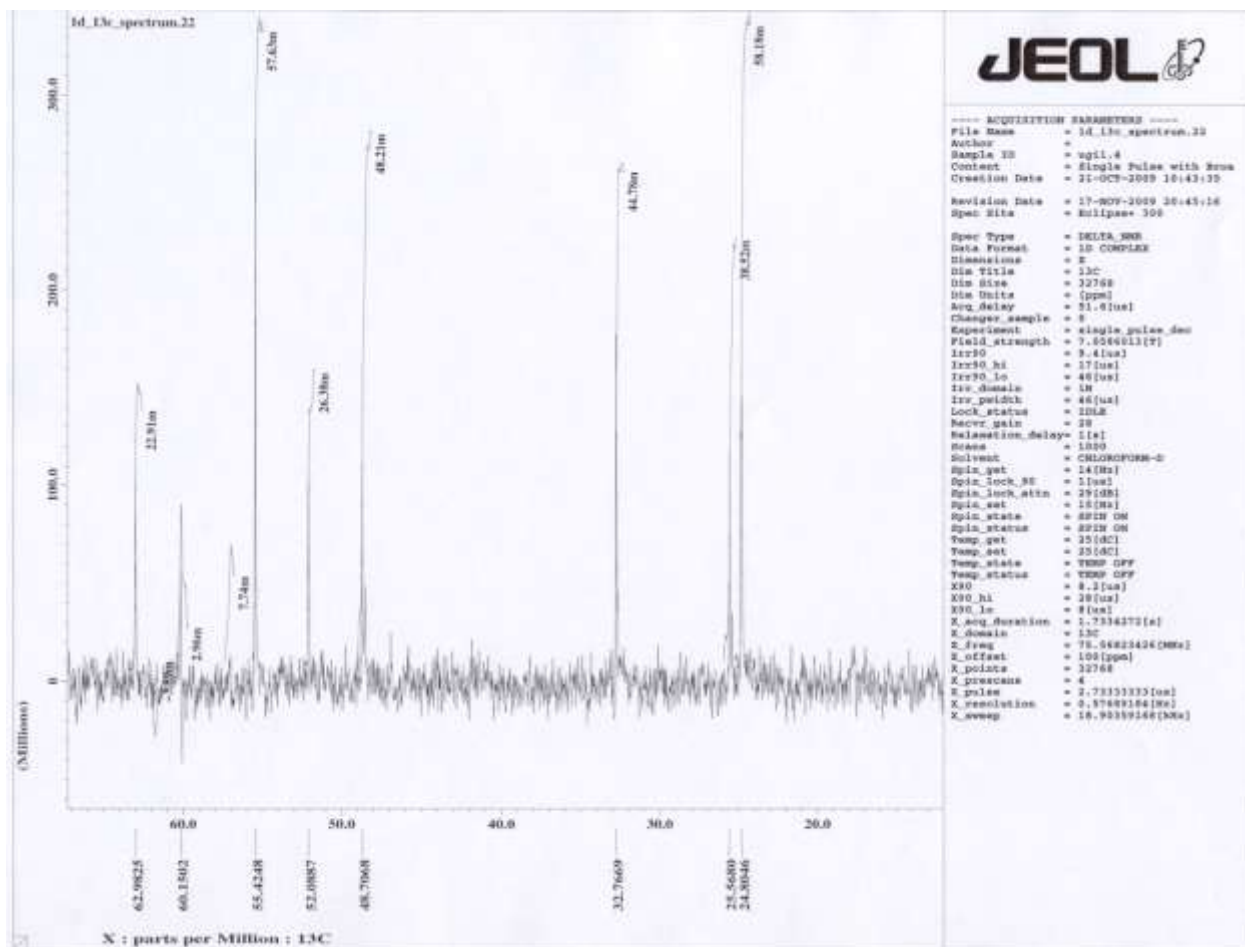
300 MHz of Ugi 233	Identity of Ugi 233	Description of Ugi 233	500 MHz of Ugi 173G	Description of Ugi 173G	300 MHz of Ugi 234	Identity of Ugi 234	Description of Ugi 234
24.8046	C6	Cyclohexyl (CH ₂)	13.1	t-Butyl	24.7282	C6	Cyclohexyl (CH ₂)
25.568	C5,C7	Cyclohexyl (CH ₂)	19.7	t-Butyl	25.4764	C5,C7	Cyclohexyl (CH ₂)
32.7669	C4,C8	Cyclohexyl (CH ₂)	30.8	t-Butyl	32.6524	C4,C8	Cyclohexyl (CH ₂)
48.7068	C3	Cyclohexyl (CH)	39	t-Butyl	48.9053	C3	Cyclohexyl (CH)
52.0887	C24	Benzylic (CH ₂)	51.4	Benzylic (CH ₂)	52.3559	C38	Benzylic (CH ₂)
55.4248	C65,C66	Methoxy (CH ₃)	54.8	Methoxy (CH ₃)			
60.1502					60.8602	C22	Hexafluoro ring (CH) (Chiral Proton)
62.9825	C22	Dimethoxy ring (CH)	62.3	Dimethoxy			

		(Chiral Proton)		ring (CH)			
76.7314	CDCl3	Solvent	76.74	Solvent	76.6932	CDCl3	Solvent
77.1513	CDCl3	Solvent	77	Solvent	77.1131	CDCl3	Solvent
77.5788	CDCl3	Solvent	77.25	Solvent	77.5406	CDCl3	Solvent
82.1516	C30	Triple Bonded Adjacent to Carbonyl	81.4	Triple Bonded Adjacent to Carbonyl	81.3805	C30	Triple Bonded Adjacent to Carbonyl
91.343	C29	Triple Bonded Adjacent to Ring	91.4	Triple Bonded Adjacent to Ring	92.5415	C29	Triple Bonded Adjacent to Ring
101.0306	C37???	Aromatic Carbons	100.6	Aromatic Carbons	119.7875		Aromatic Carbons
107.7486		Aromatic Carbons			121.1921		Aromatic Carbons
108.0845		Aromatic Carbons			122.2762		Aromatic Carbons
120.2913		Aromatic Carbons	119.6	Aromatic Carbons	124.8107		Aromatic Carbons
127.2001		Aromatic Carbons	126.6	Aromatic Carbons	127.2688		Aromatic Carbons
			126.7	Aromatic Carbons			
127.2994		Aromatic Carbons	127.3	Aromatic Carbons	127.7727		Aromatic Carbons
128.3147		Aromatic Carbons	127.7	Aromatic Carbons	128.6735		Aromatic Carbons
128.5285		Aromatic Carbons	127.8	Aromatic Carbons	129.8644		Aromatic Carbons
128.6506		Aromatic Carbons	128.2	Aromatic Carbons	130.7042		Aromatic Carbons
130.2614		Aromatic Carbons	128.3	Aromatic Carbons	131.4065		Aromatic Carbons
			130.1	Aromatic Carbons	131.8493		Aromatic Carbons
132.5592		Aromatic Carbons	130.3	Aromatic Carbons	132.292		Aromatic Carbons
132.8493		Aromatic Carbons	135.5	Aromatic Carbons	132.6814		Aromatic Carbons
136.4221		Aromatic Carbons	137.1	Aromatic Carbons	136.4602		Aromatic Carbons

137.5366		Aromatic Carbons	137.5	Aromatic Carbons	136.903		Aromatic Carbons
138.2		Aromatic Carbons	156.2	Carbonyl Adjacent to Triple Bond			
156.2248		Carbonyl Adjacent to Triple Bond	156.3	Carbonyl Adjacent to Triple Bond	156.2248	C31	Carbonyl Adjacent to Triple Bond
160.9045			160.4				
161.1106			160.6				
167.6301			168.78		166.5918	C1	Carbonyl Adjacent to Amide
167.8286			168.945				

Table 2: Carbon NMR Results and identification with HyperChem Atom Labeling of Ugi 233, Ugi 173G, and Ugi234





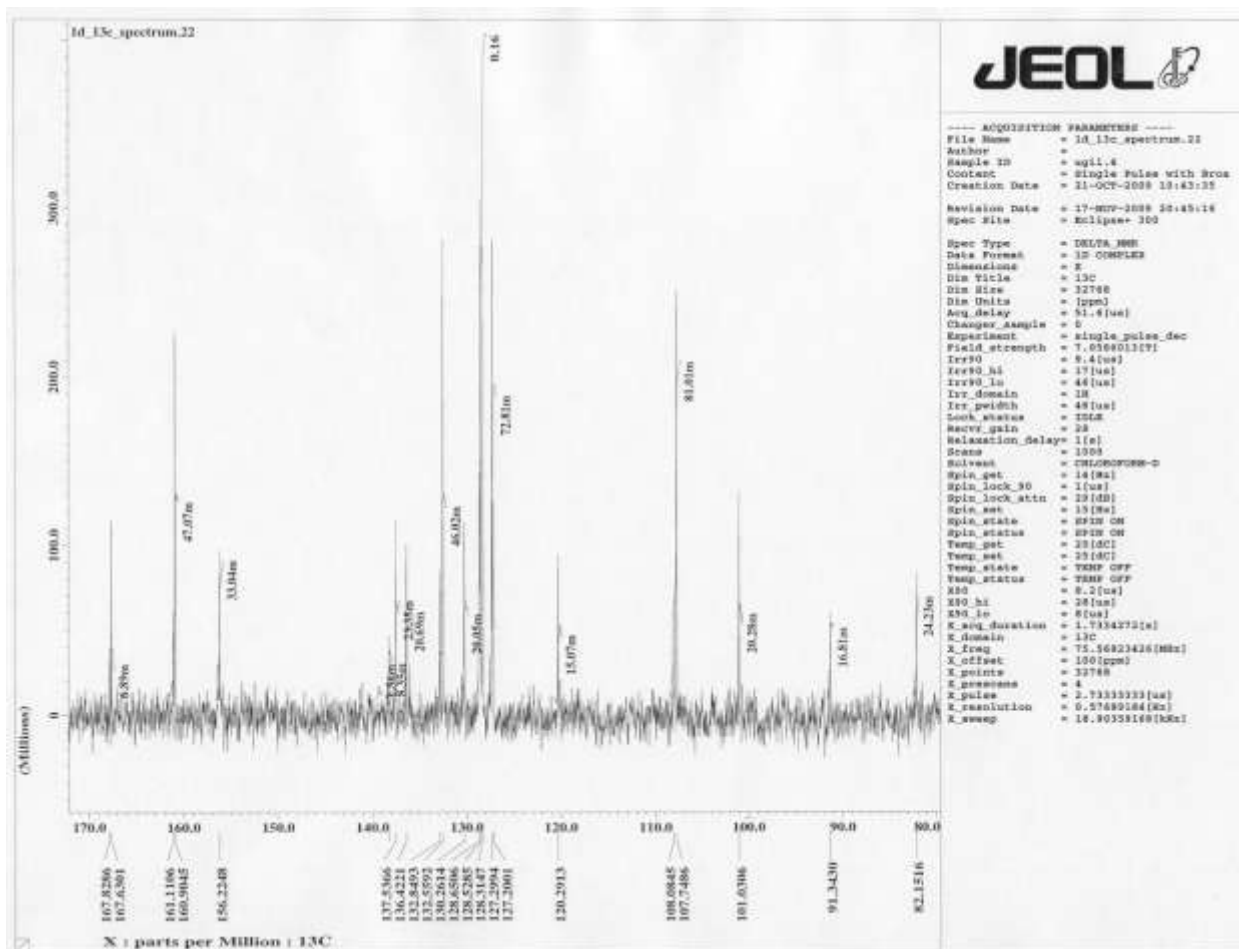
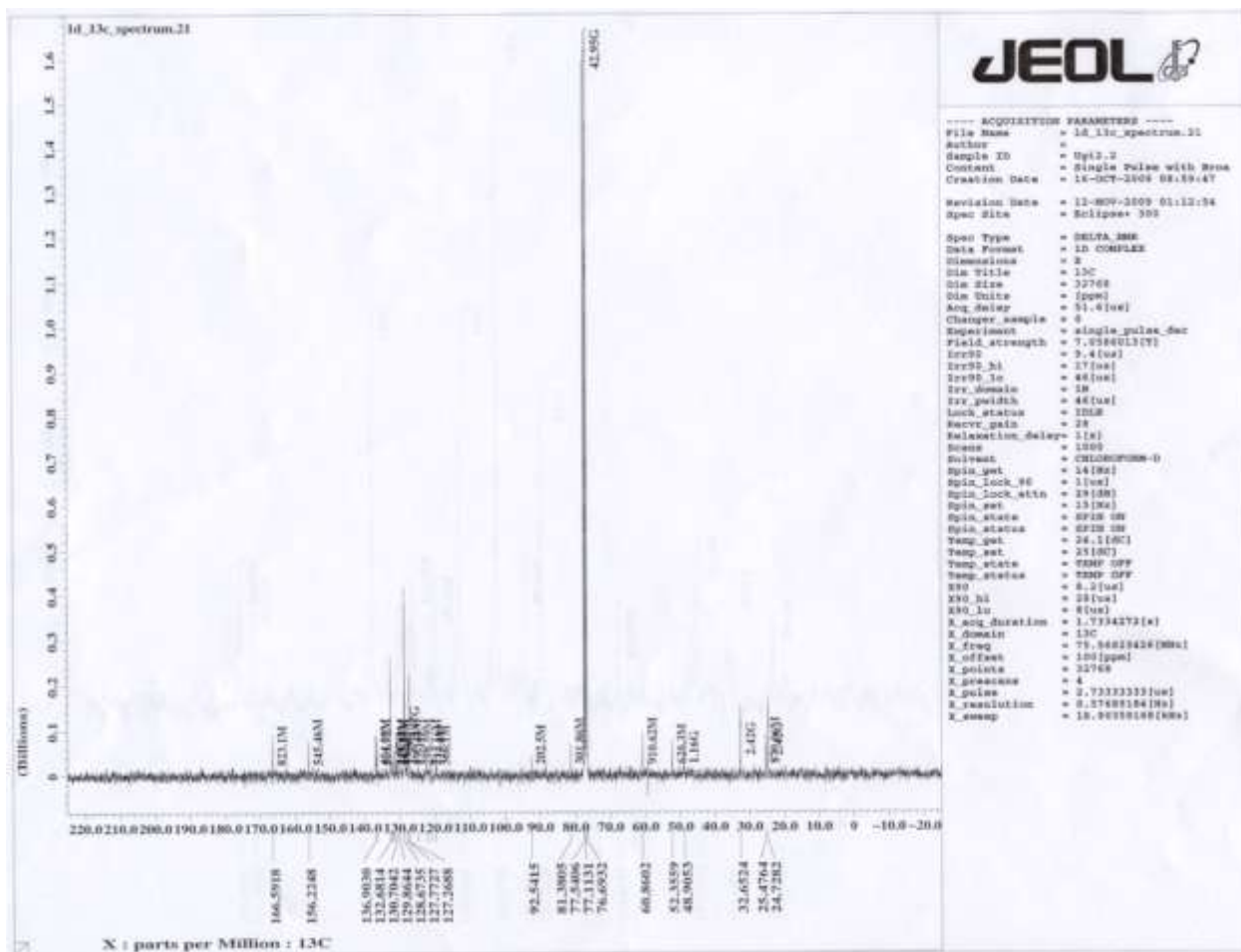
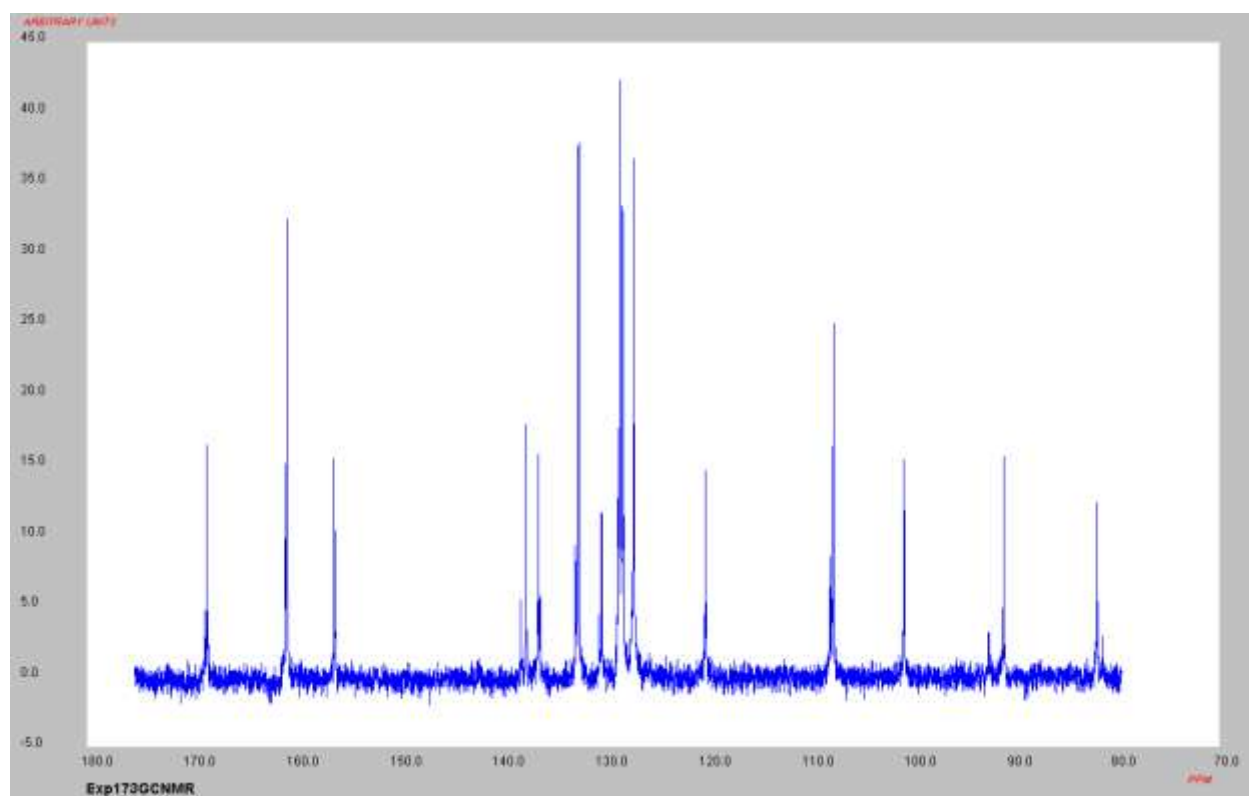
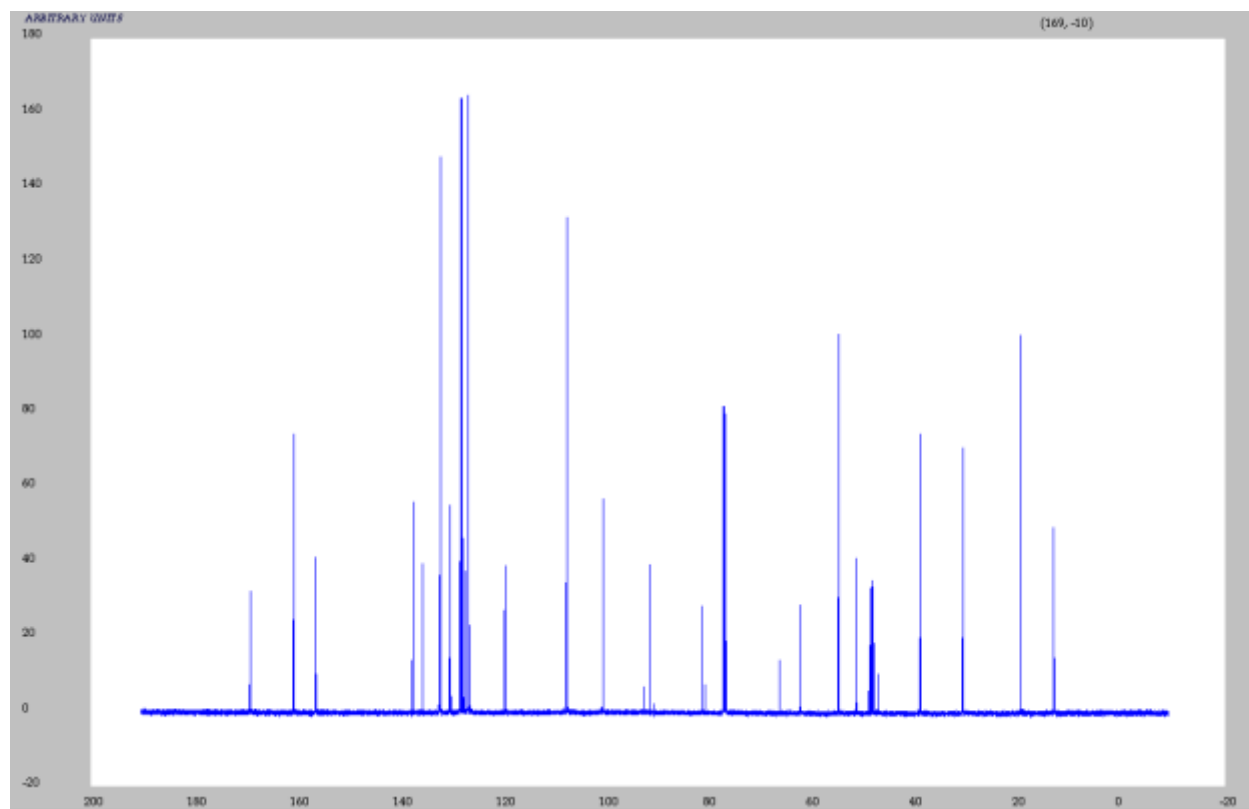


Figure 4: C NMR of Ugi 233





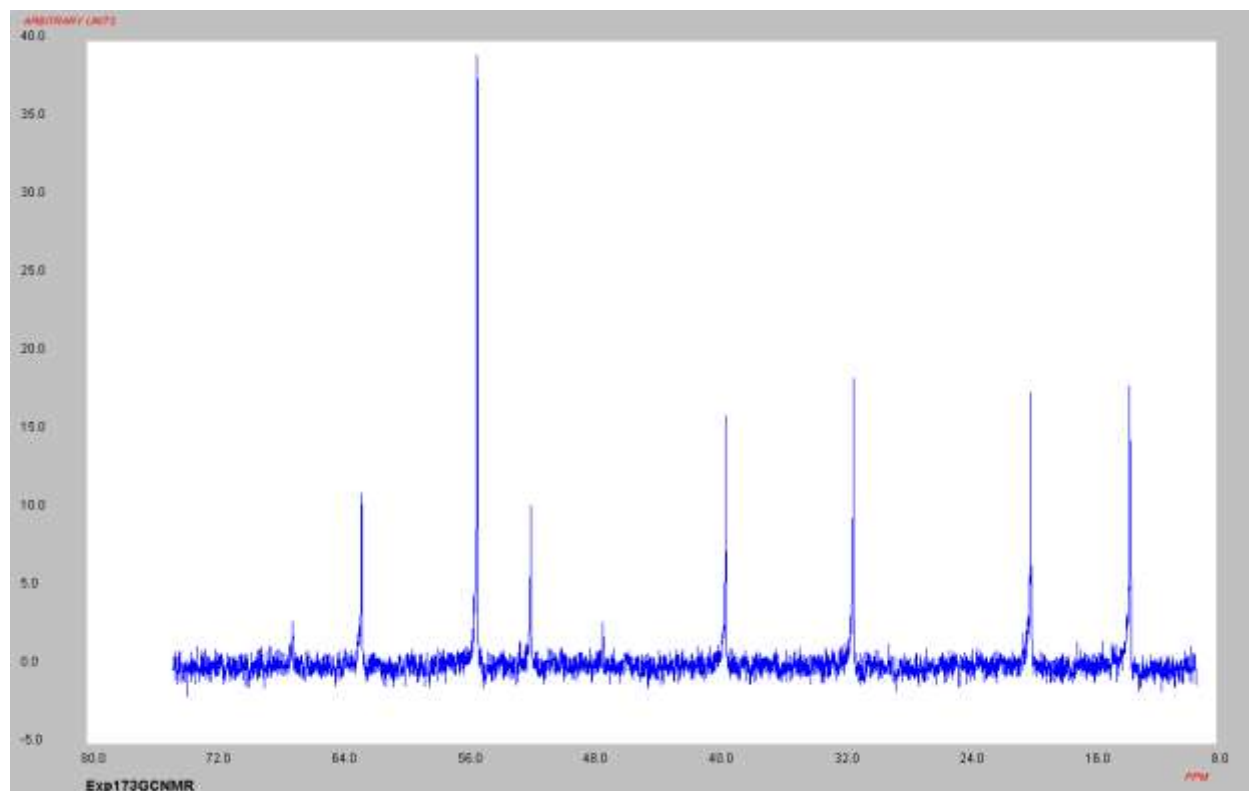


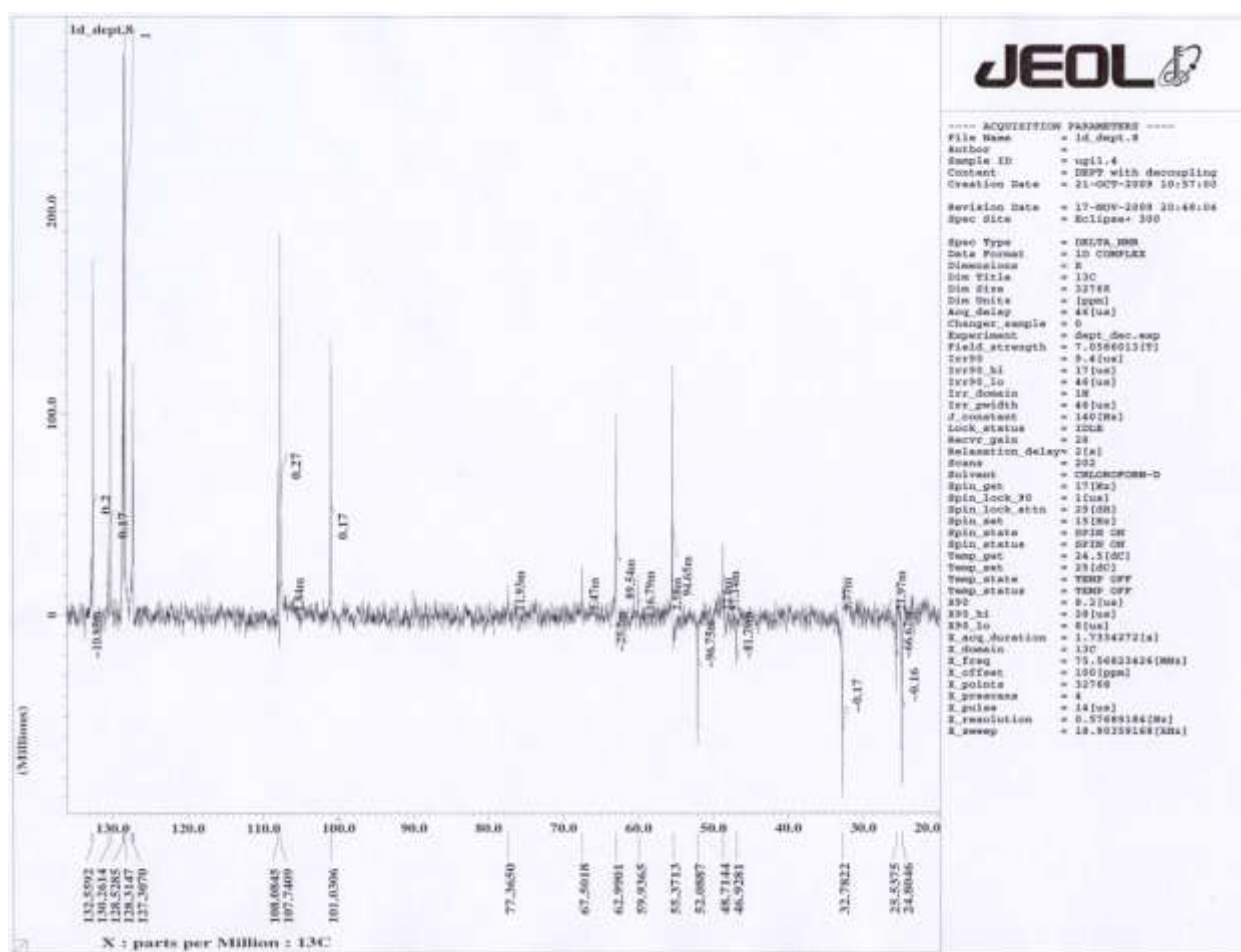
Figure 6: C NMR of Ugi 173G

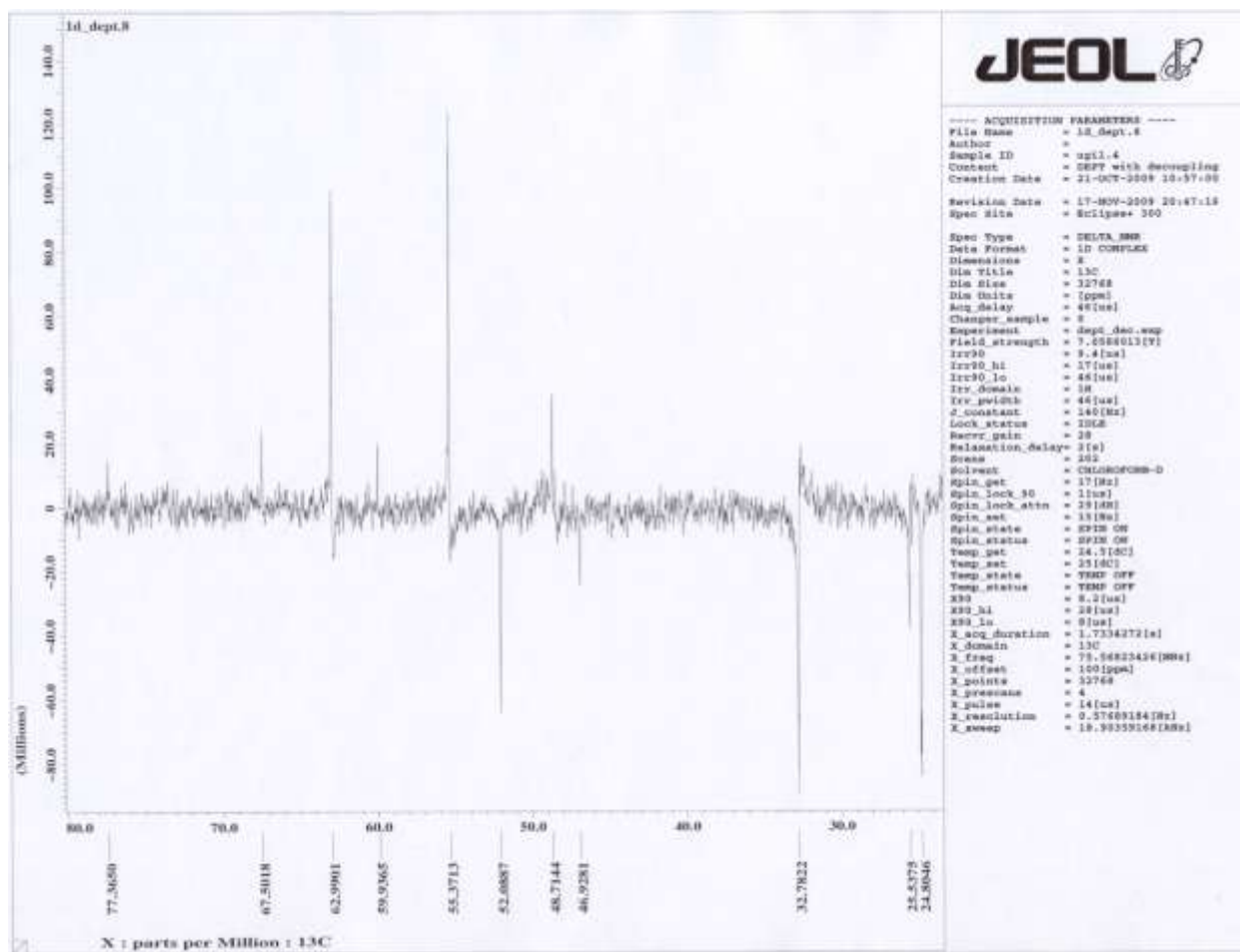
C13 Chemical Shift (ppm)	Direction of DEPT 135 peak	DEPT135 (ppm)	DEPT90 (ppm)	chshf proton 1 (ppm)	chshf proton 2 (ppm)
24.8046	down	24.8046	Distorted	1.6	
25.568	down	25.5375			
32.7669	down	32.7822			
	down	46.9281			
48.7068	up	48.7144			
52.0887	down	52.0887		4.89	5.01
55.4248	up	55.3713		3.65	
60.1502	up	59.9365		dimethoxy	

62.9825	up	62.9901		5.7	
	up	67.5018		6.1	
76.7314					
77.1513					
77.5788	up	77.3650		7.26	
82.1516					
91.343					
101.0306	up	101.0306		6.32	
107.7486	up	107.7409		6.42	
108.0845	up	108.0845		6.40	
120.2913					
127.2001	up	127.2001		7.19	
127.2994	up	127.3070			
	up	127.5208			
128.3147	up	128.3147		7.20	
128.5285	up	128.5285		7.24	
128.6506	up	128.6506		7.28	
130.2614	up	130.2614			
	up	130.5591			
132.5592	up	132.5592		7.35	
132.8493	up	132.8493			
136.4221					
137.5366					
138.2					

156.2248					
160.9045					
161.1106					
167.6301					
167.8286					

Table 3: Other Carbon/Proton NMR Spectra in Relation to Carbon 13 Spectra of Ugi 233





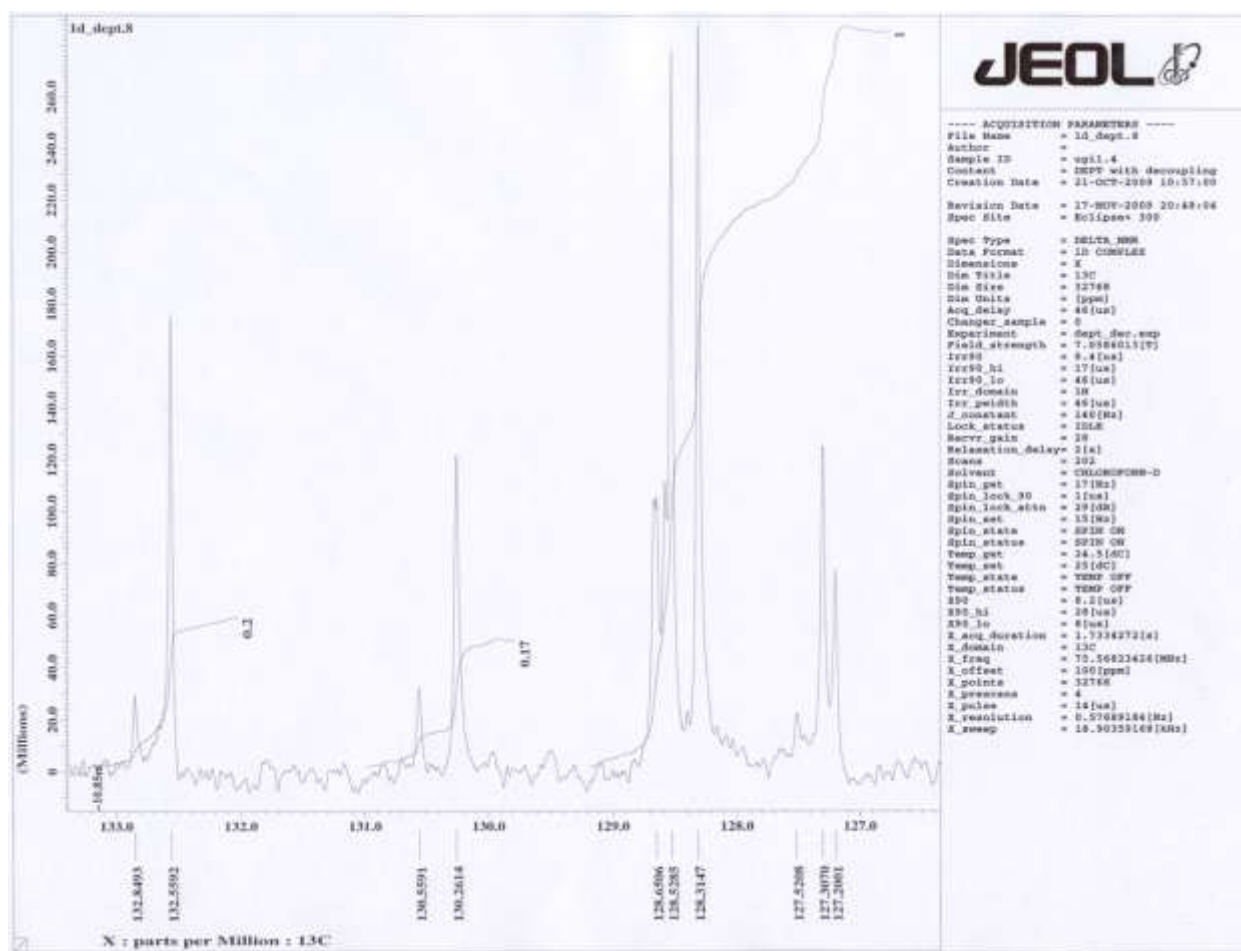


Figure 7: DEPT135 of Ugi 233

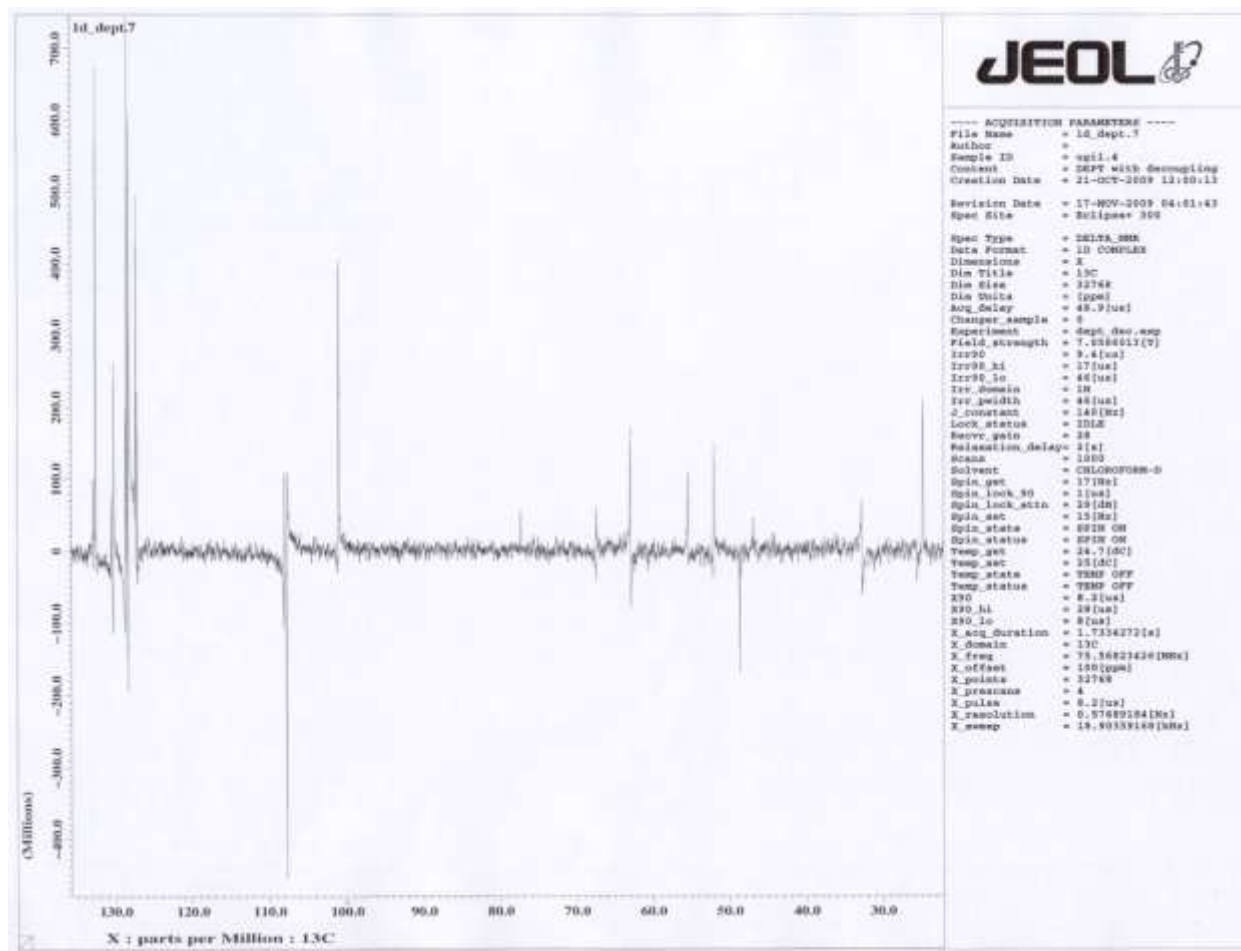


Figure 8: DEPT90 of Ugi 233

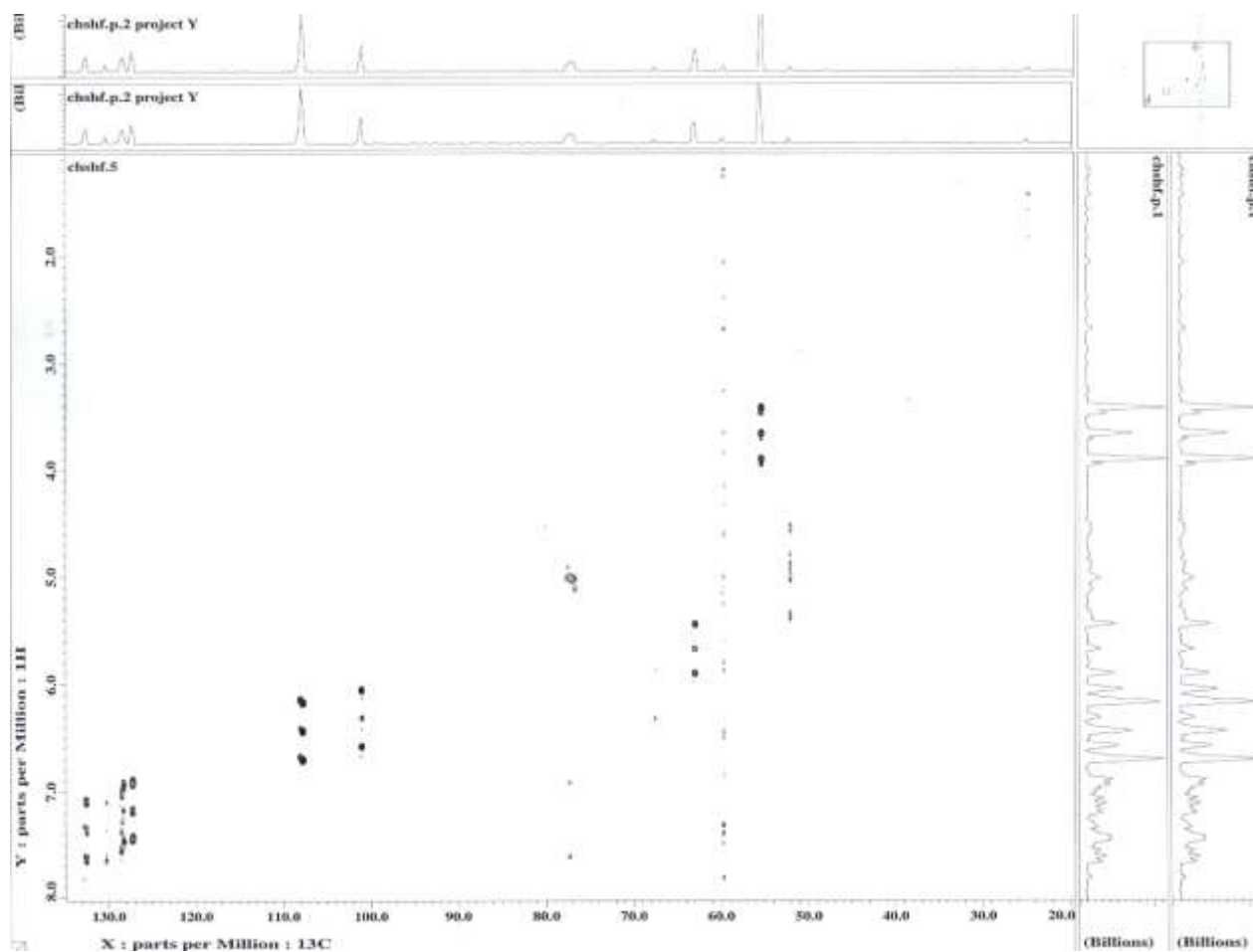


Figure 9: chshf of Ugi 233

C13 Chemical Shift (ppm)	Direction of DEPT 135 peak	DEPT135 (ppm)	DEPT90 (ppm)	chshf proton 1 (ppm)	chshf proton 2 (ppm)
24.7282	down	24.7359	24.7359	1.55	
25.4764	down	25.4382	25.4458	1.62	
32.6524	down	32.5990	32.5990	1.19	1.95
48.9053	up	48.9129	48.9053	3.82	
52.3559	down	52.3559	52.3635	5.12	
60.8602	up	60.8602	60.8678	6.05	
	up	65.4635	65.4559		

76.6932					
77.1131	up	77.2429	77.3192	7.27	
77.5406					
81.3805					
92.5415					
119.7875					
121.1921					
122.2762	up	122.2227	122.2685	7.70	
124.8107					
127.2688	up	127.2688	127.2688	7.14	
127.7727	up	127.7727	127.7650	7.225	
	up	127.9025	127.9025		
	up	128.0475	128.0475		
128.6735	up	128.6811	128.6735	7.282	
			128.7880		
			128.9331		
129.8644	up	129.8262	129.8186	7.755	
130.7042	up	130.7042	130.7042	7.485	
131.4065					
131.8493					
132.2920					
132.6814	up	132.6814	132.6814	7.462	
136.4602					
136.9030					
156.2248					

Table 4: Other Carbon/Proton NMR Spectra in Relation to Carbon 13 Spectra of Ugi 234

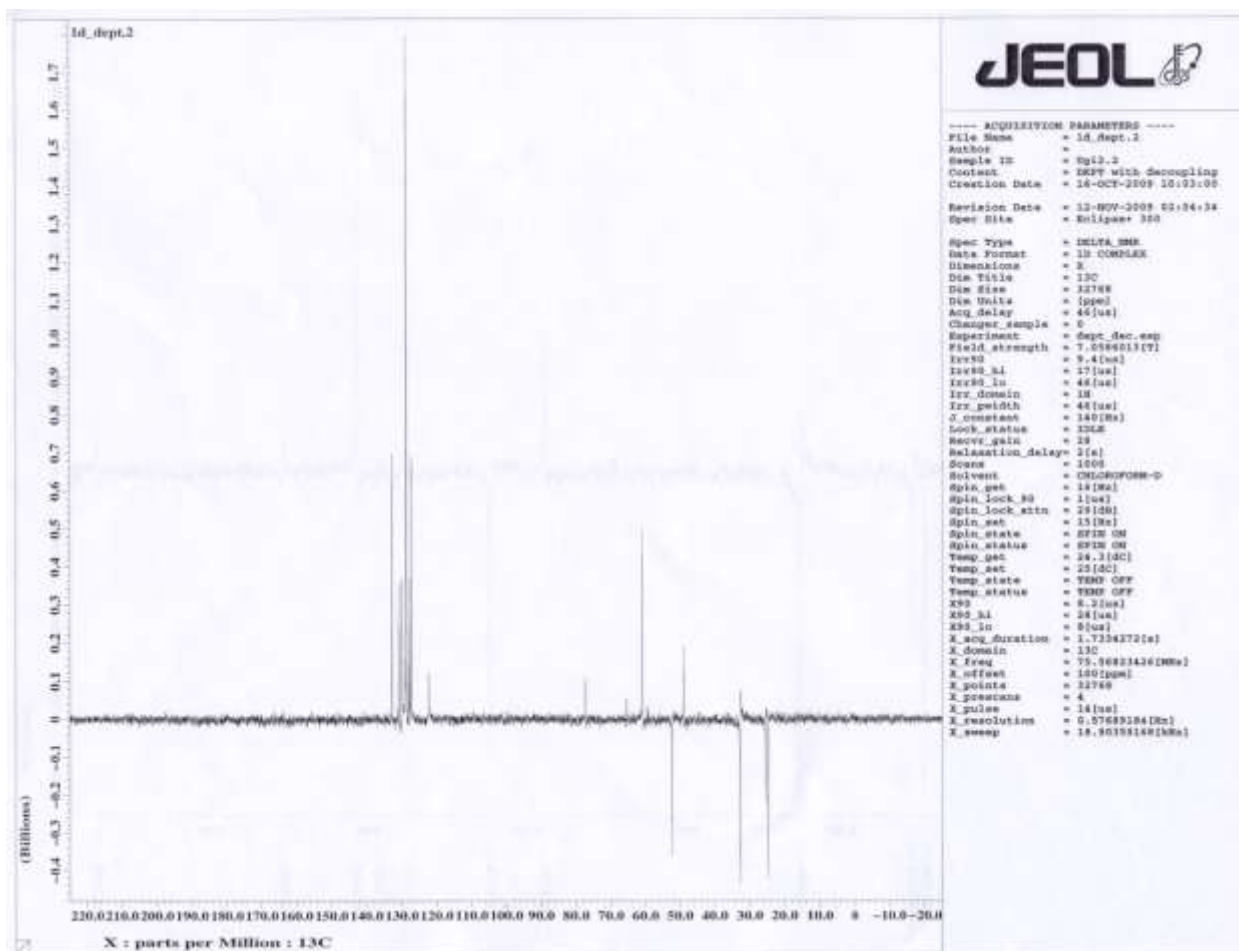
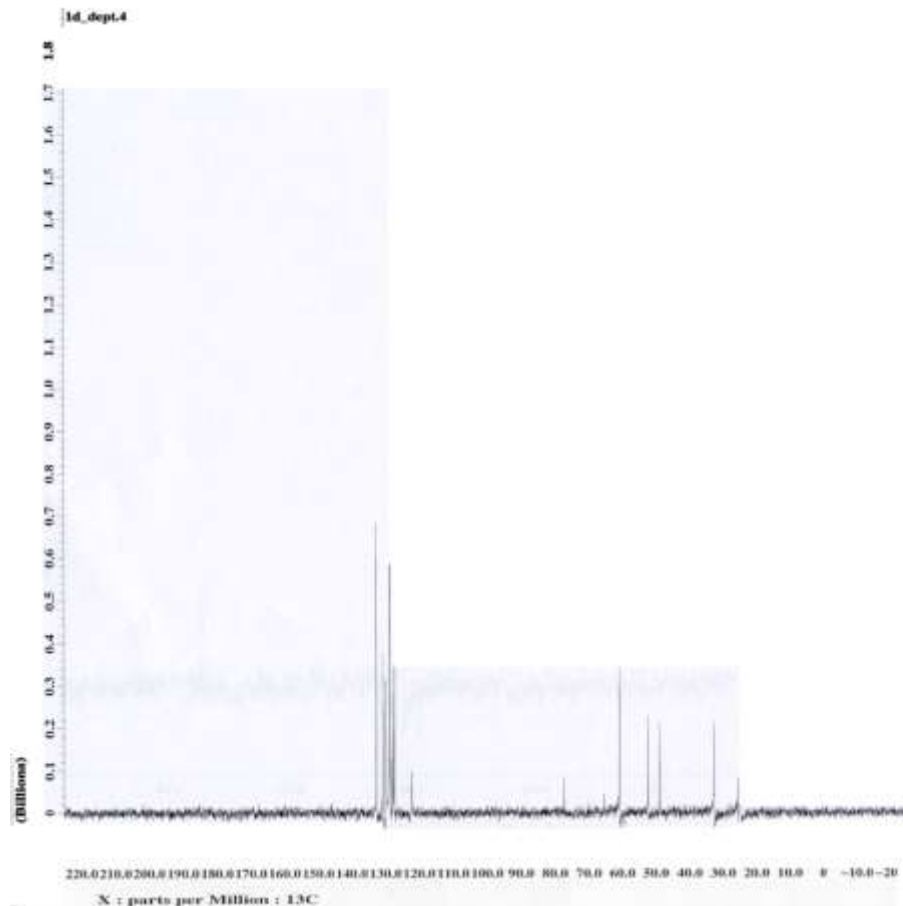


Figure 10: DEPT135 of Ugi 234



JEOL

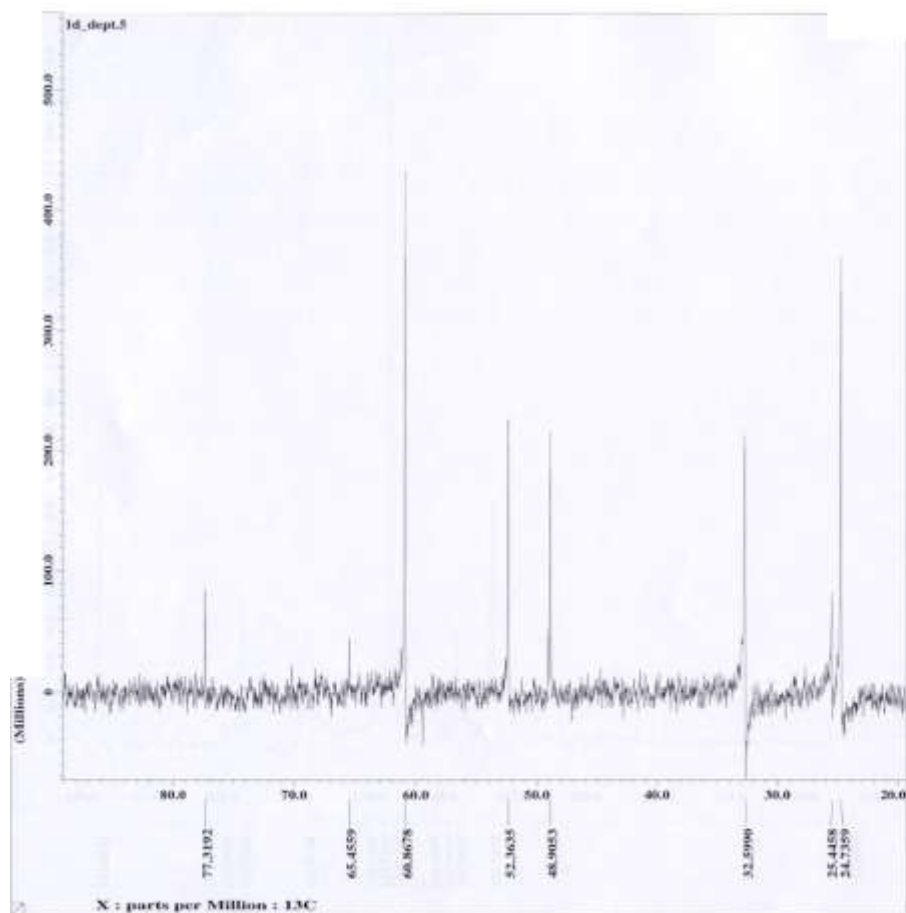
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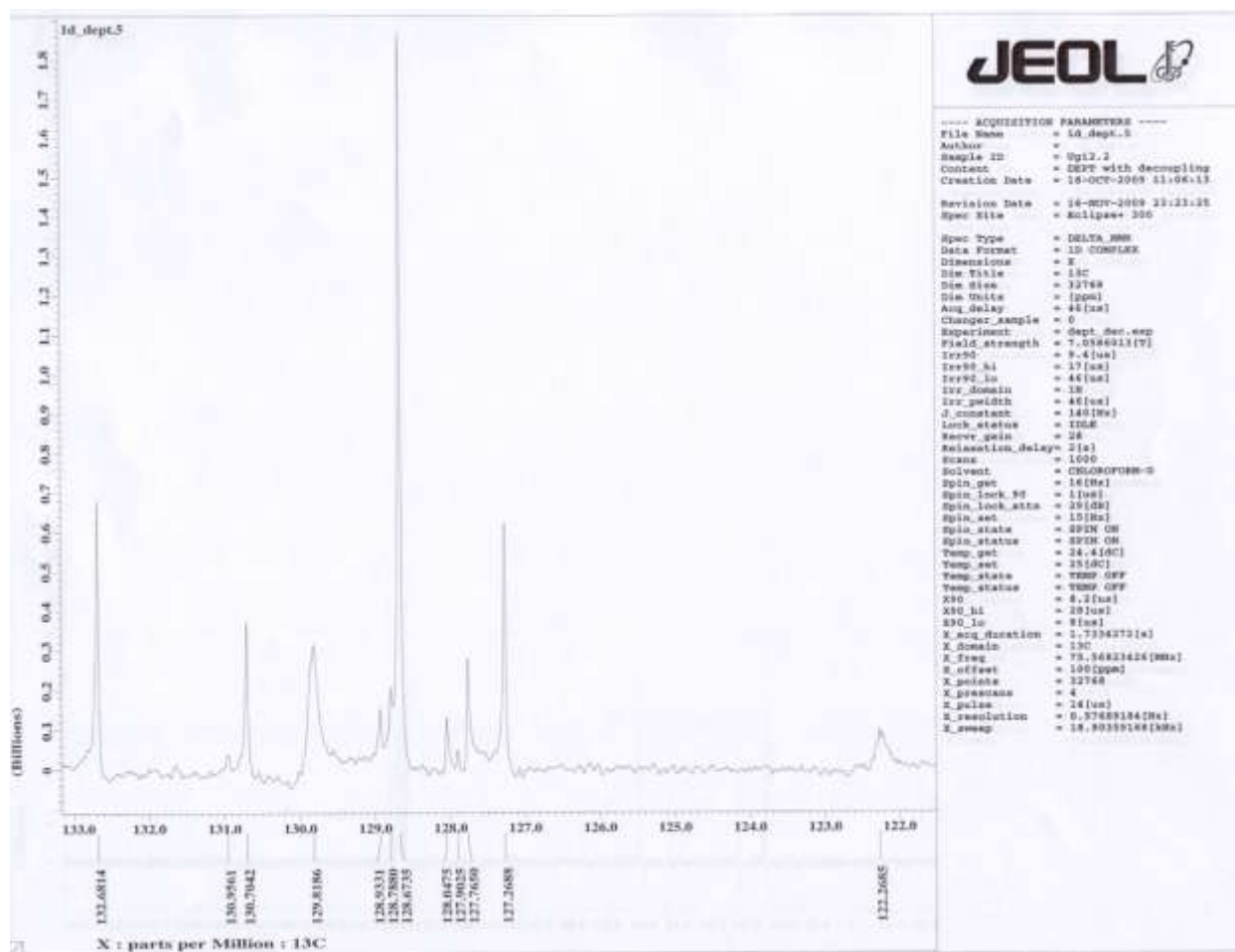


Figure 11: DEPT90 of Ugi 234

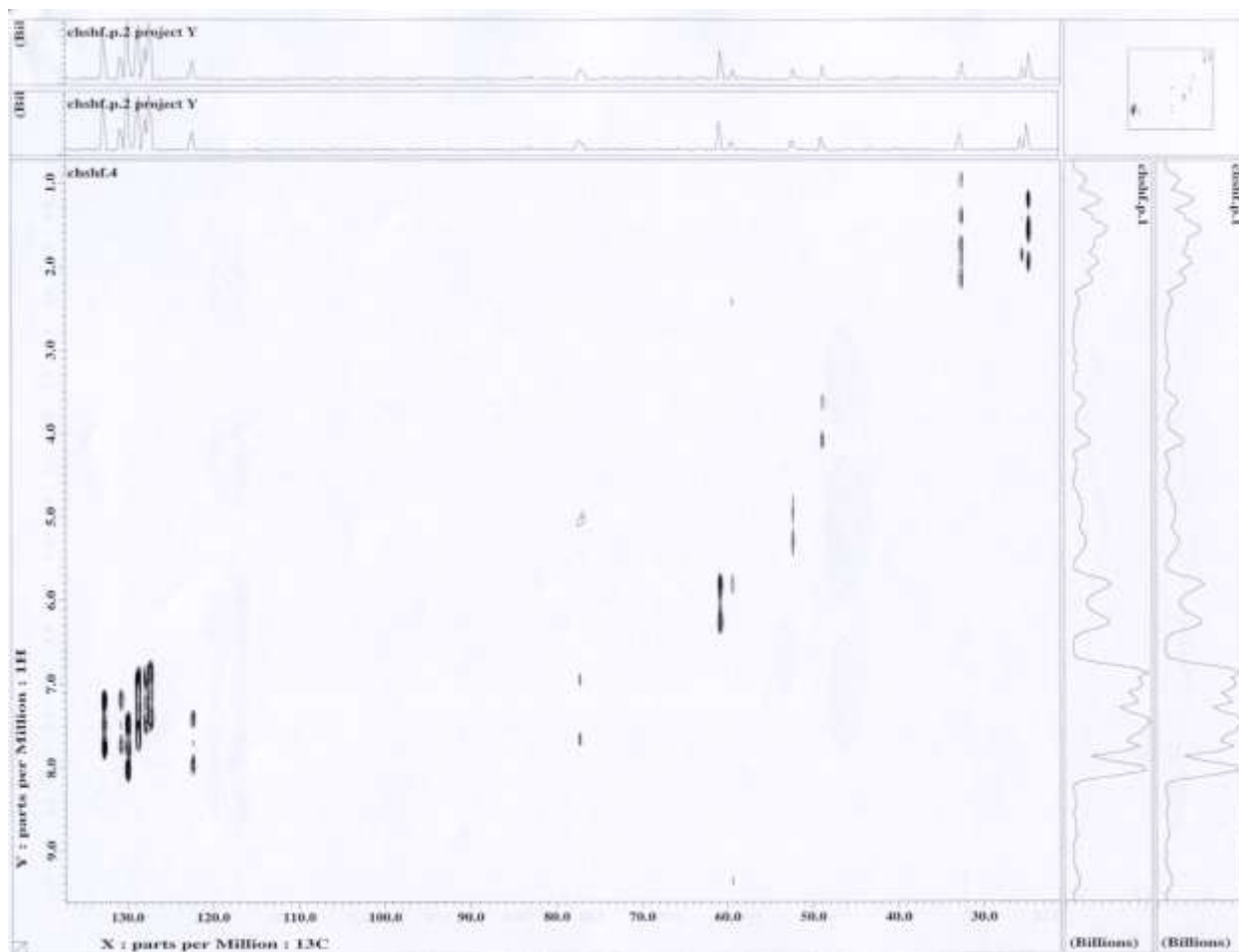
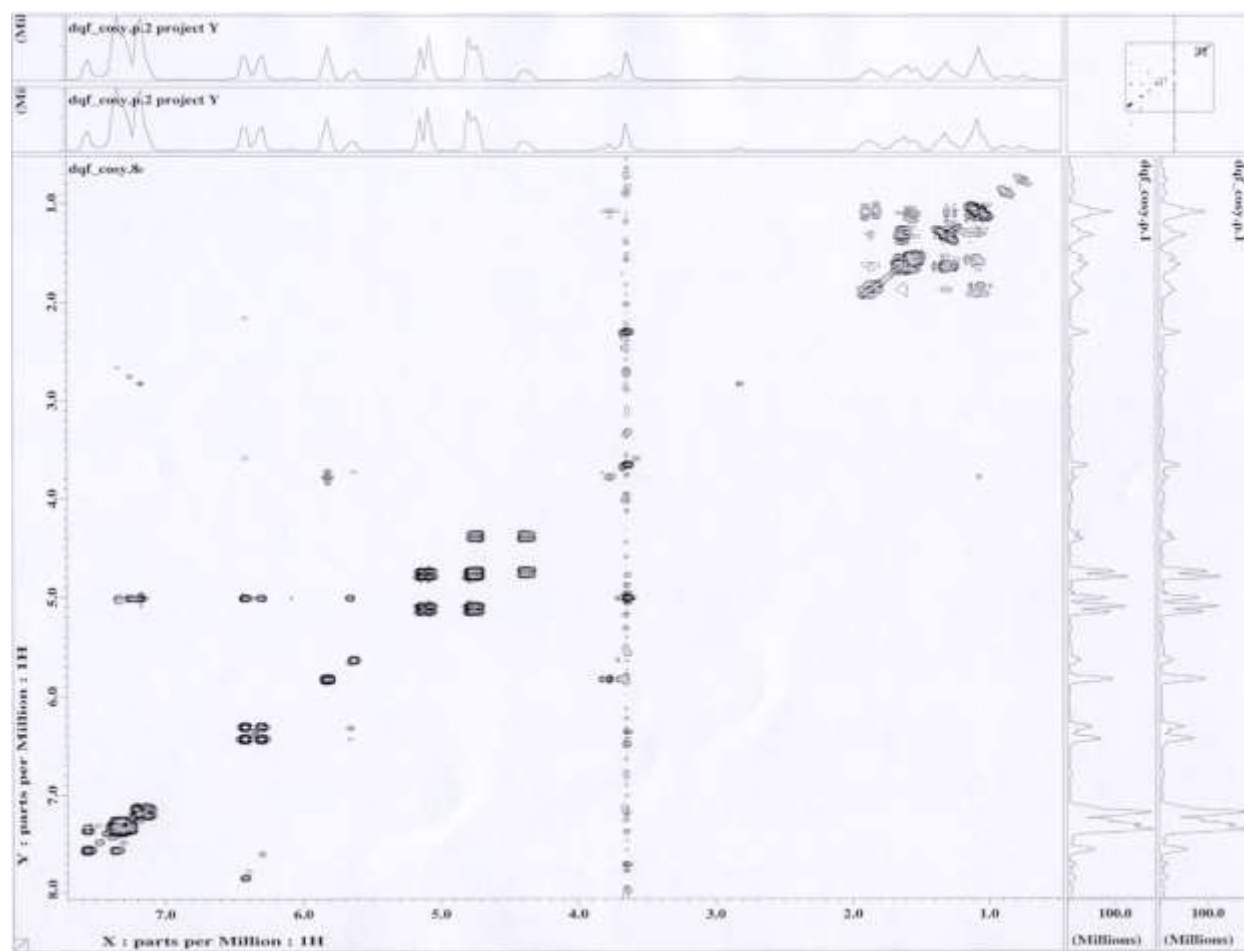


Figure 12: chshf of Ugi 234

H NMR (ppm)	COSY H1 (ppm)	COSY H2 (ppm)	NOESY H1 (ppm)	NOESY H2 (ppm)
0.65-0.98				
0.98-1.2	1.48-1.75	1.8-1.98		
1.2-1.42	1.48-1.75			
1.48-1.75	0.98-1.2	1.2-1.42		
1.8-1.98	0.98-1.2			
3.65			6.575,6.581	
3.685				

3.7-3.85				
4.335-4.4				
4.73,4.79	5.09,5.15			
5.09,5.15	4.73,4.79			
5.167,5.214	5.467,5.513		5.467,5.513	
5.467,5.513	5.167,5.214		5.167,5.214	
5.91				
5.94			6.575,6.581	6.395
6.068,6.091				
6.395			5.94	
6.694,6.4755,6.4816	6.575,6.581			
6.552				
6.575,6.581	6.694,6.4755,6.4816		3.65	5.94
7.176-7.257				
7.257-7.322	7.322-7.392			
7.322-7.392	7.5167,7.5377	7.257-7.322	7.5167,7.5377	
7.5167,7.5377	7.322-7.392		7.322-7.392	

Table 5: Other Proton/Proton NMR Spectra in Relation to Proton Spectra of Ugi 233



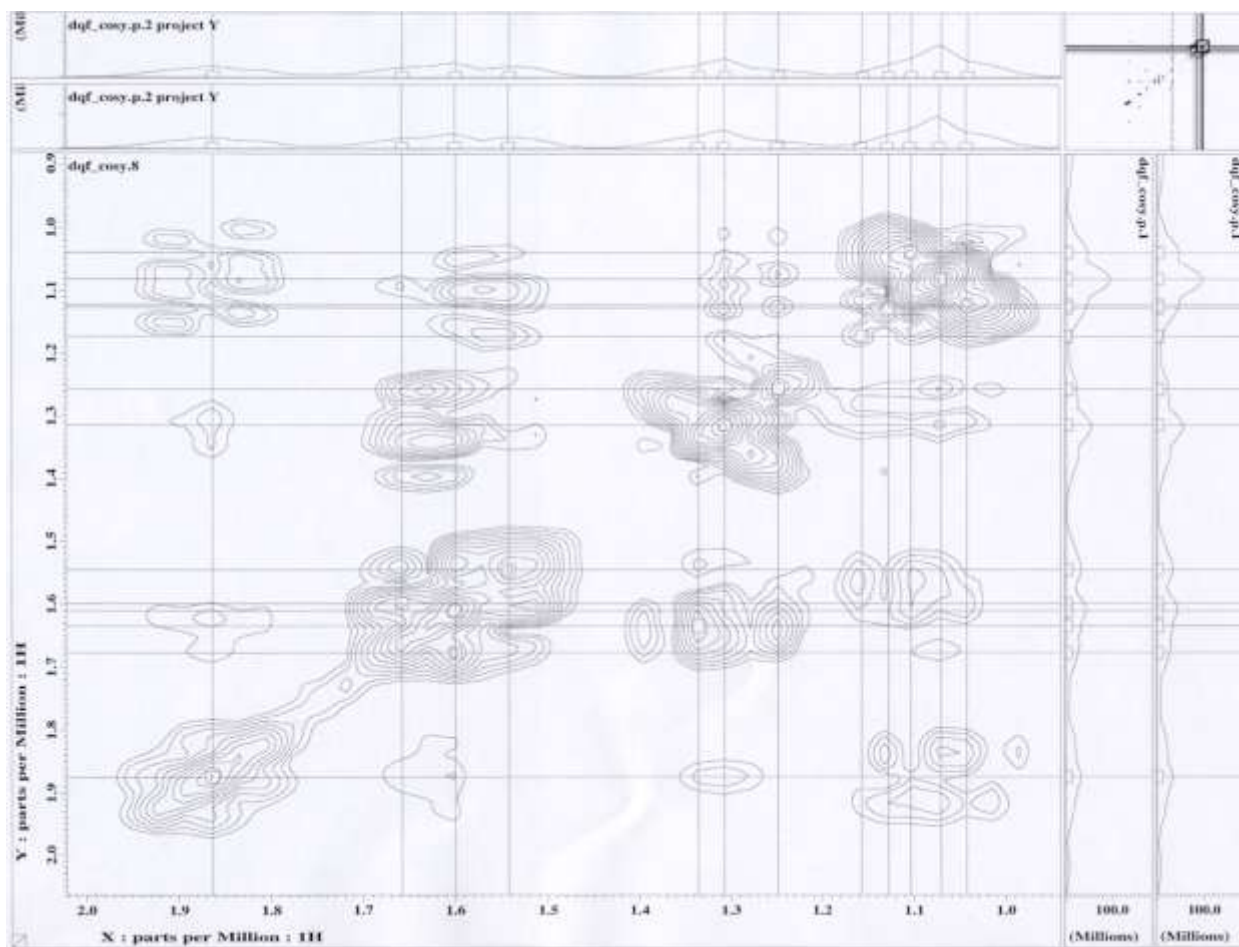


Figure 13: COSY of Ugi 233

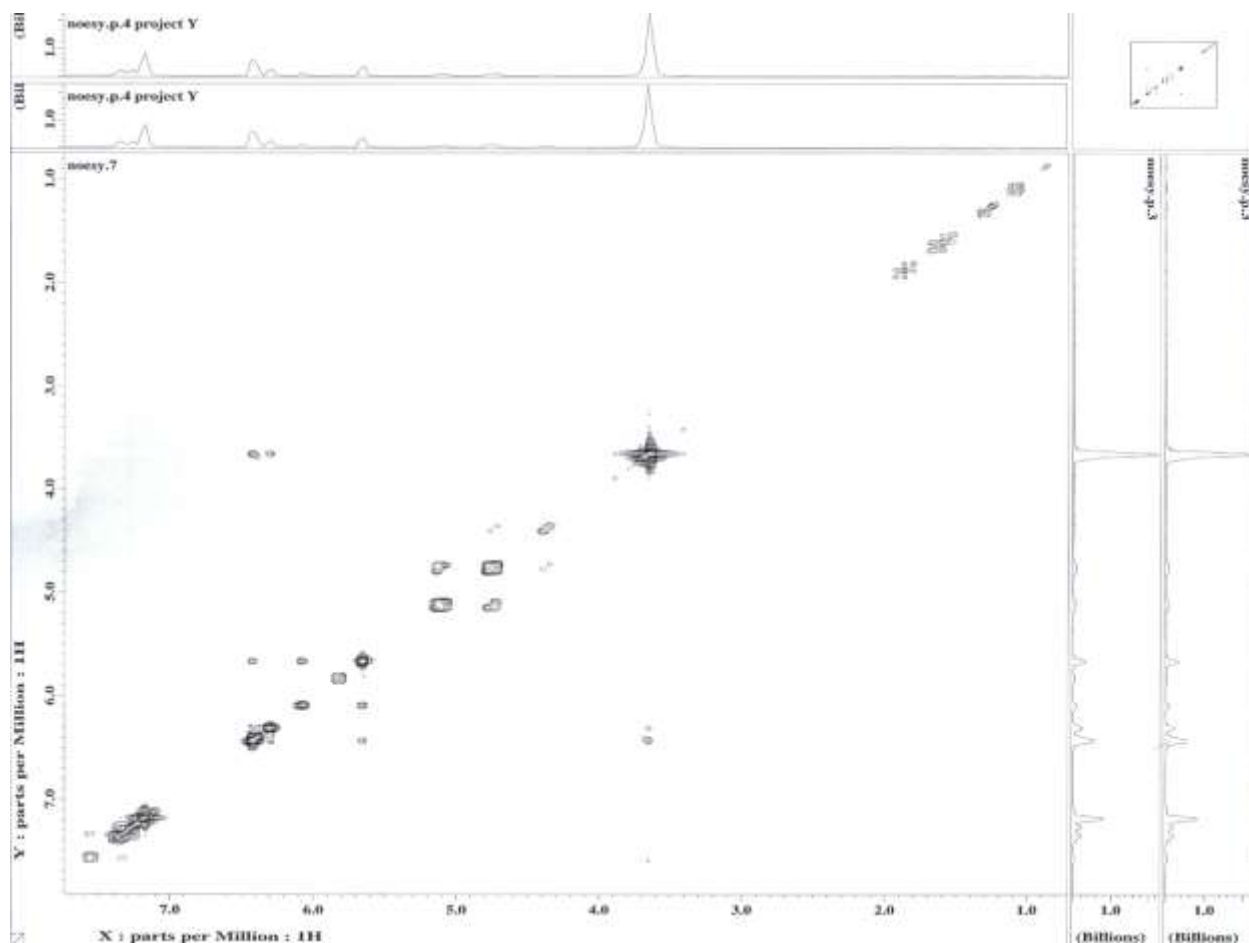
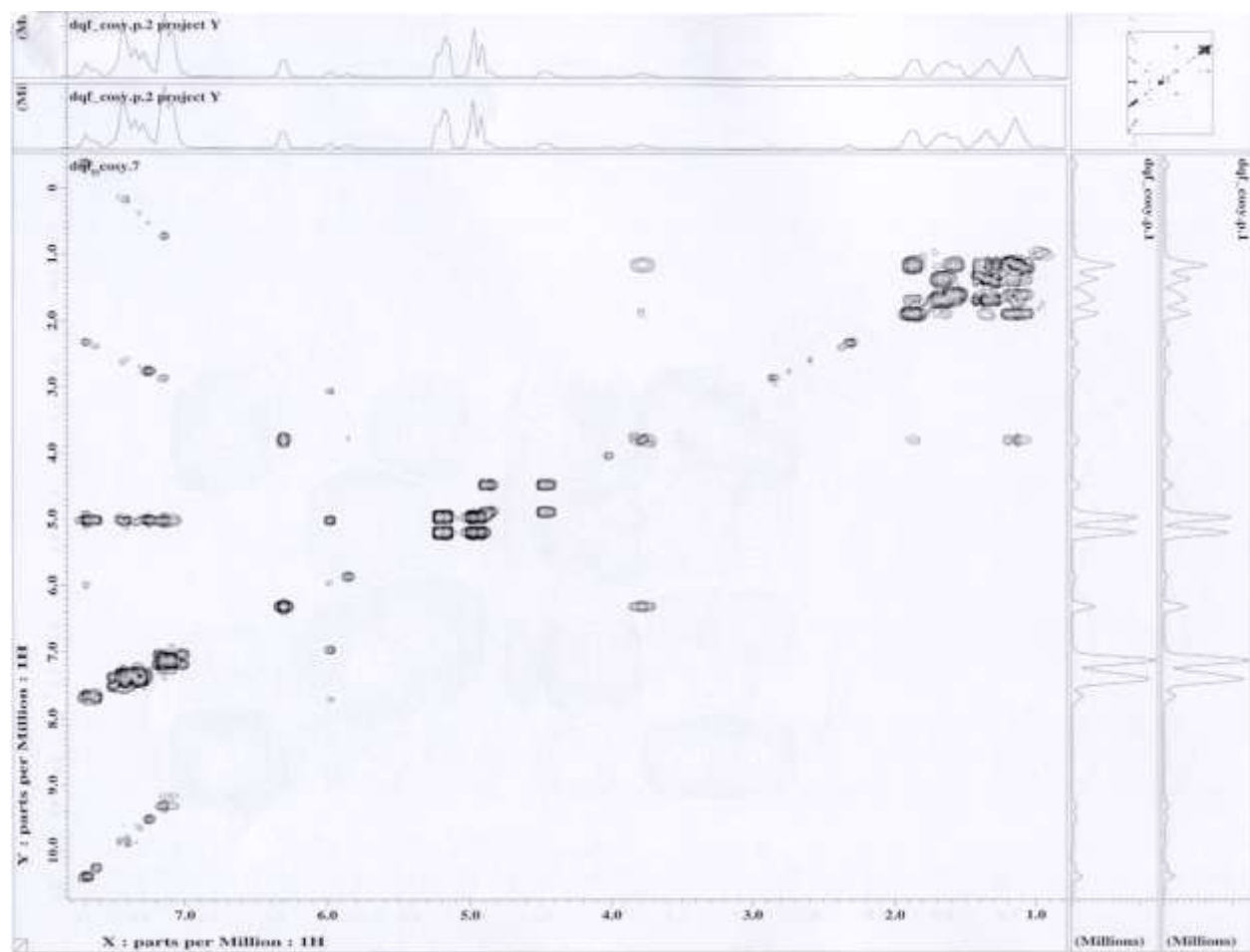


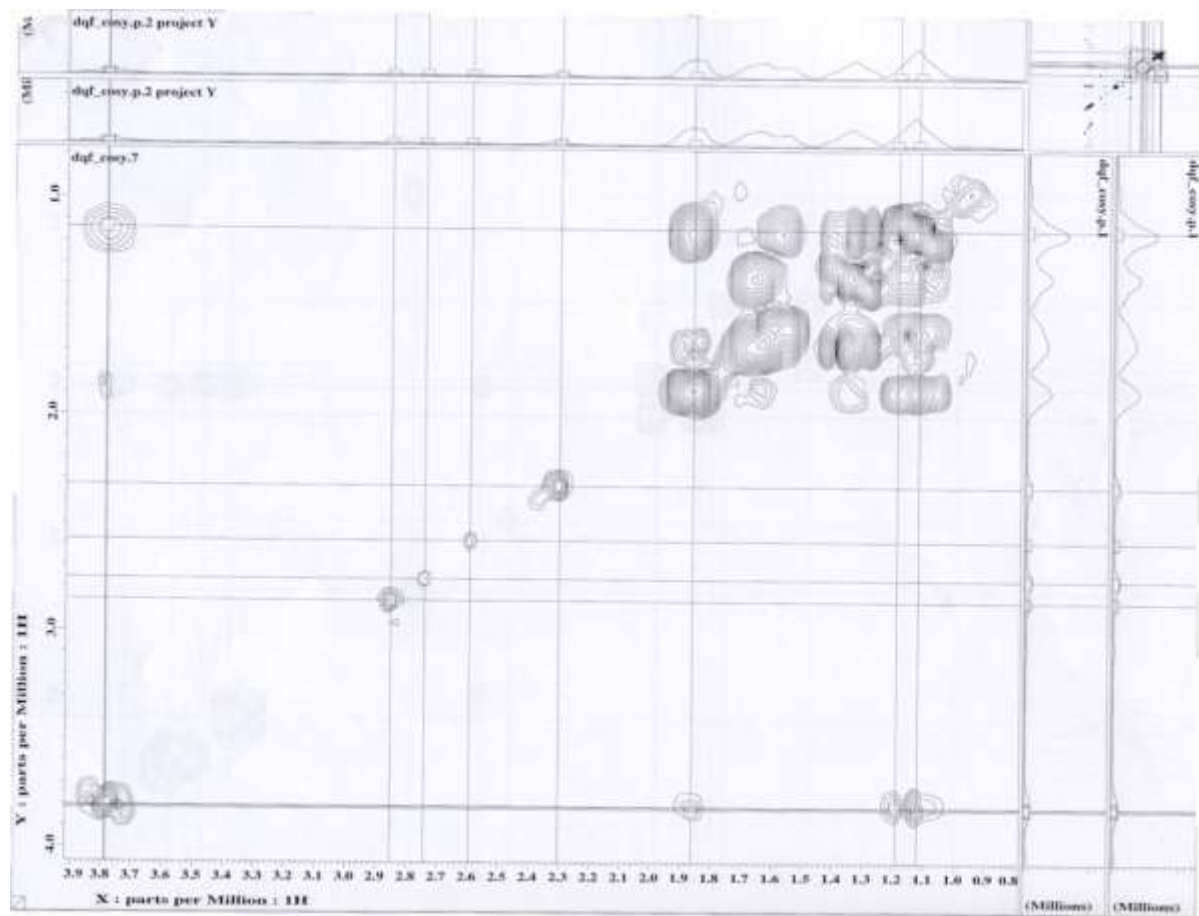
Figure 14: NOESY of Ugi 233

H NMR	COSY H1 (ppm)	COSY H2 (ppm)	COSY H3 (ppm)	NOESY H1 (ppm)	NOESY H2 (ppm)
0.88-1.1					
1.01-1.25	1.8-2.0	4.29-4.44			
1.25-1.45	1.5-1.75				
1.5-1.75	1.25-1.45				
1.8-2.0	1.01-1.25	4.29-4.44			
4.29-4.44	1.01-1.25	1.8-2.0	6.461,6.484		
4.911,4.953	5.259,5.303				

5.259,5.303	4.911,4.953				
5.313,5.359	5.566,5.520			5.566,5.520	
5.566,5.520	5.313,5.359			5.313,5.359	
6.085,6.108					
6.198				7.654	6.327
6.327				6.198	
6.461,6.484	4.29-4.44				
7.028-7.227					
7.26					
7.276-7.432					
7.447-7.488					
7.569					
7.624					
7.654				6.198	

Table 6: Other Proton/Proton NMR Spectra in Relation to Proton Spectra of Ugi 234





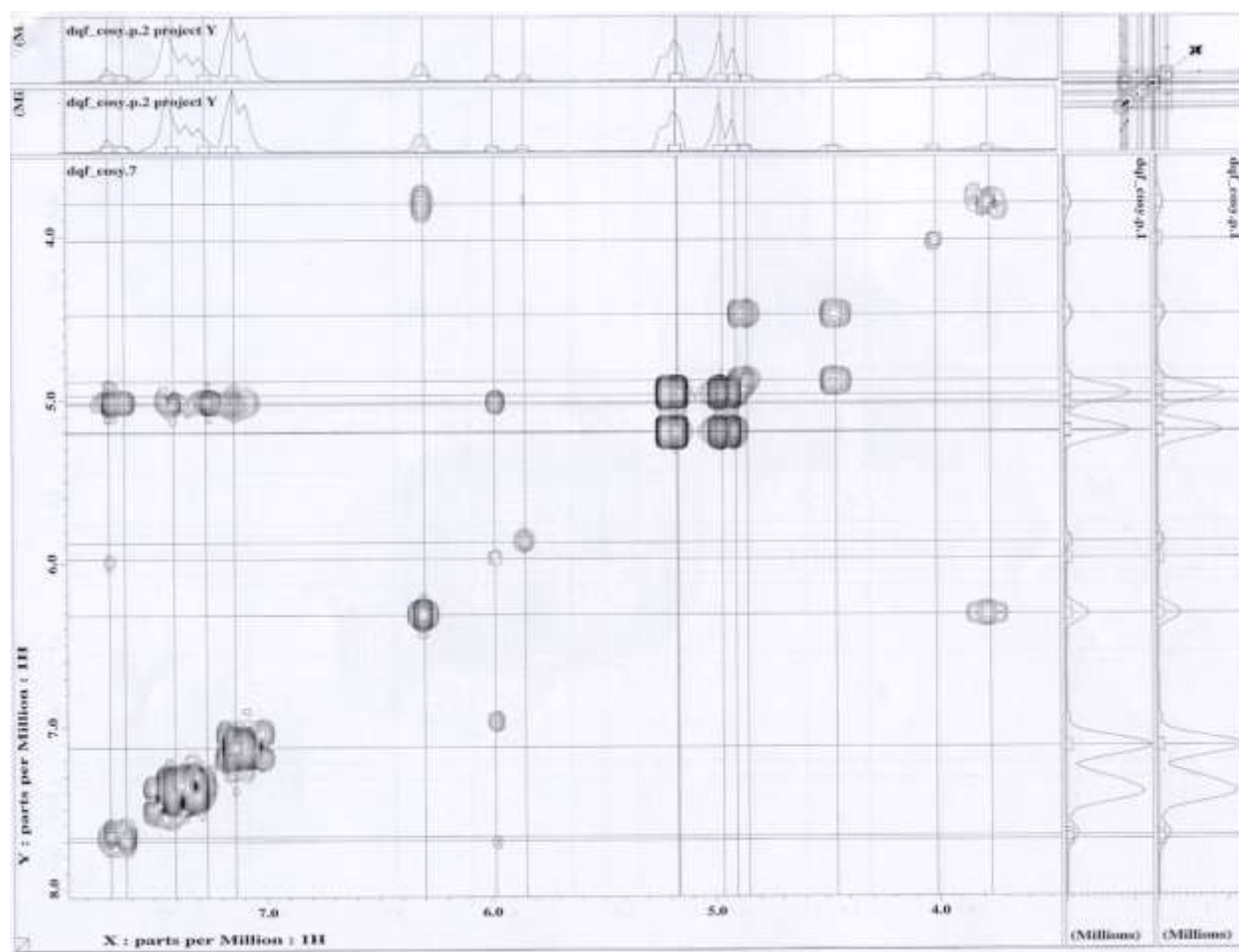


Figure 15: COSY of Ugi 234

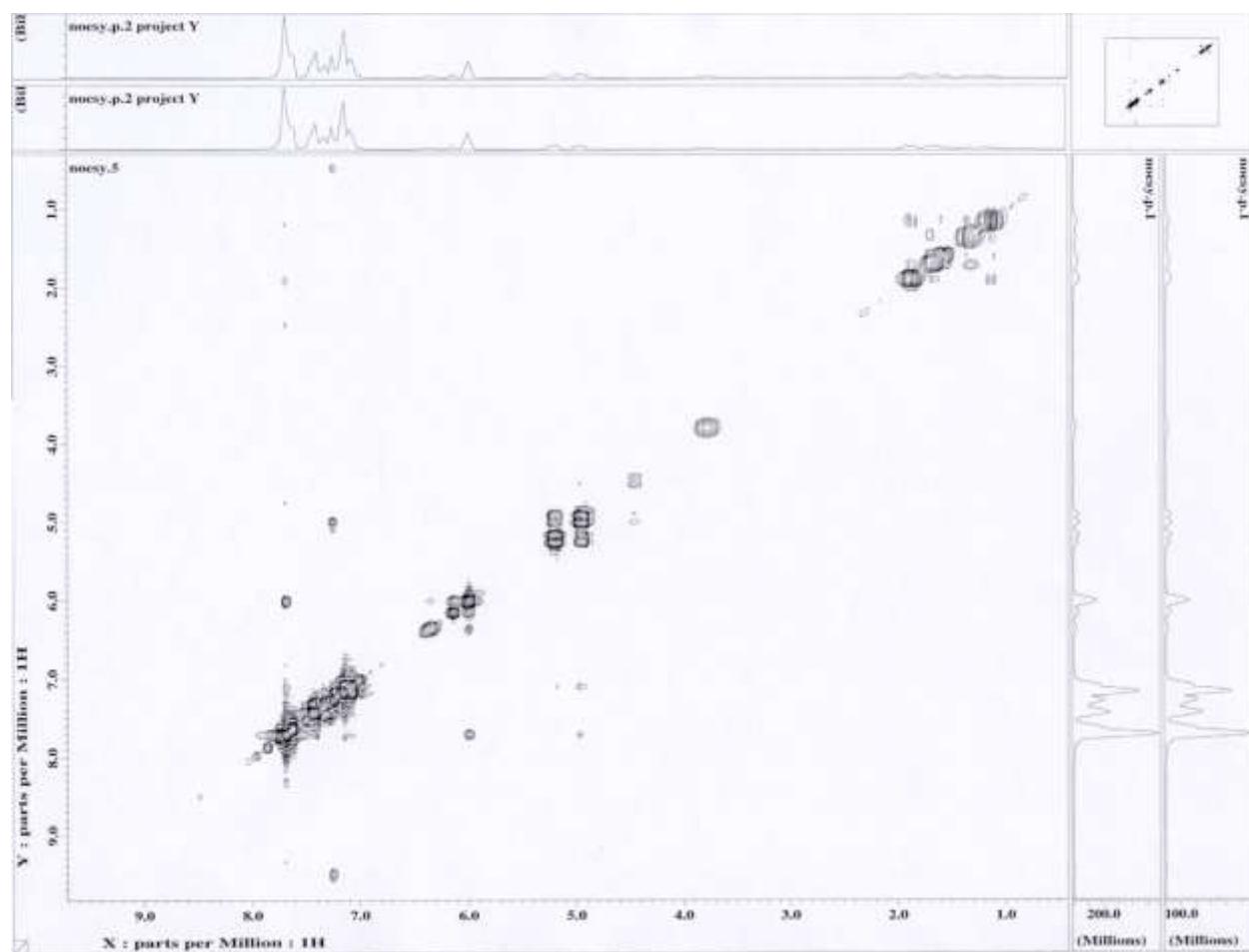


Figure 16: NOESY of Ugi 234

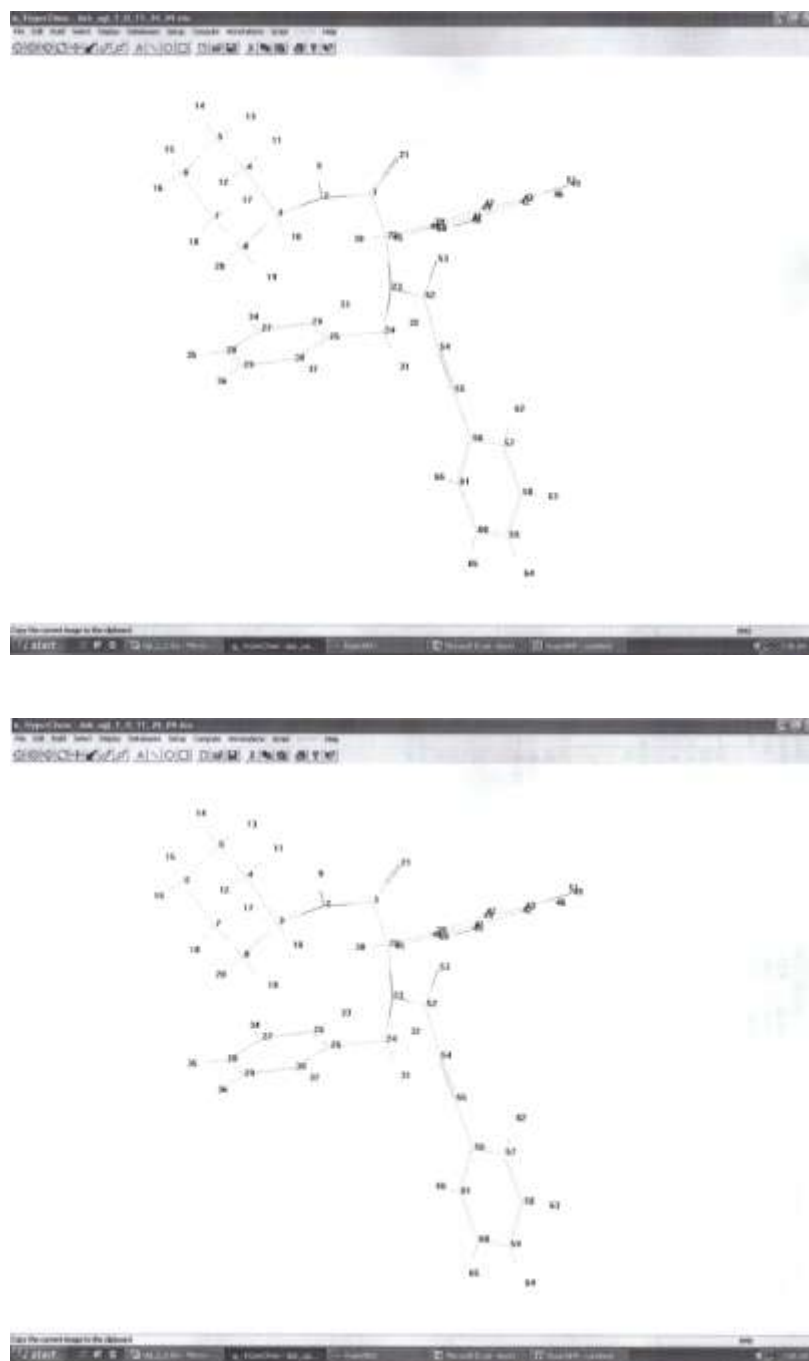


Figure 17: Structure and Labeling of Ugi 233 Open

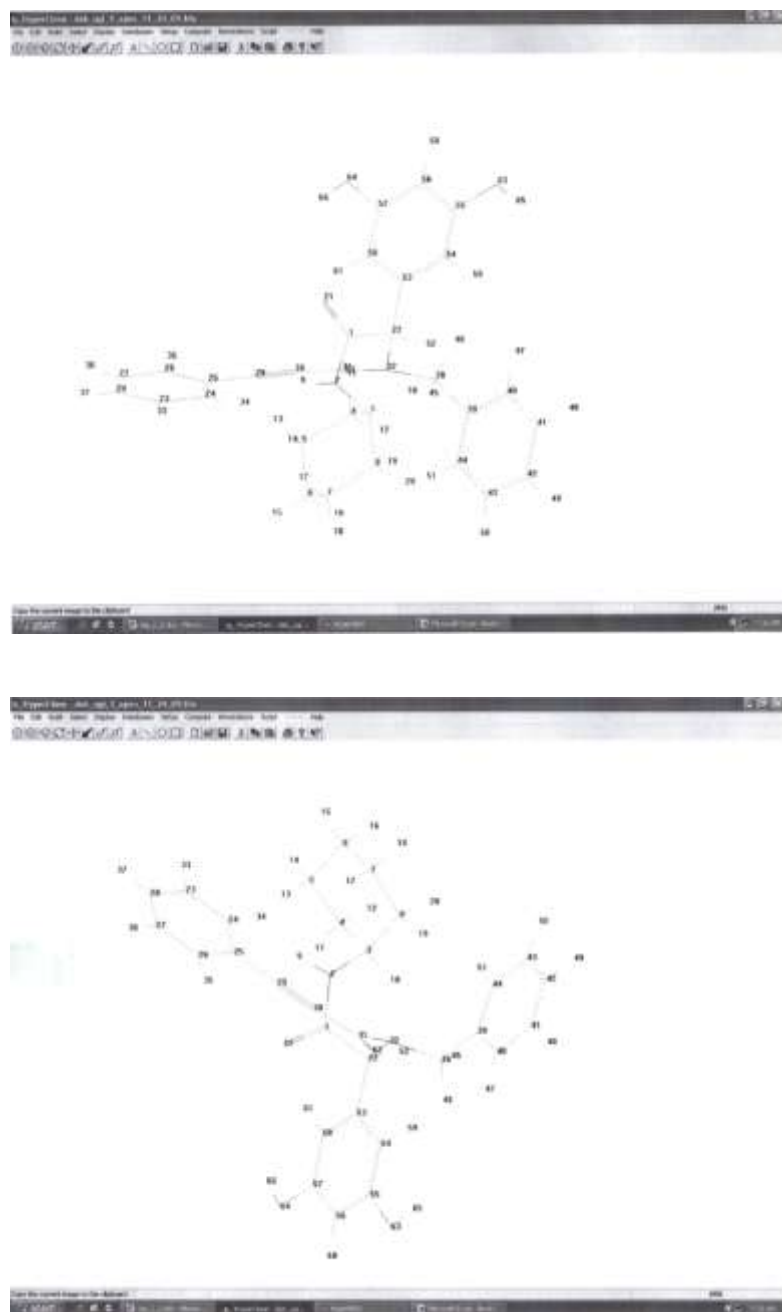


Figure 18: Structure and Labeling of Ugi 233 Closed

Index (C)	Shift (C)	Index (CMD) 0.1 ps	Shift (CMD) 0.1 ps	Index (O)	Shift (O)	Index (OMD) 0.2 ps	Shift (OMD) 0.2 ps
19H	0.693	15H	0.693	19H	0.896	14H	-0.606

20H	1.044	20H	0.85	20H	0.904	13H	0.22
16H	1.188	18H	0.983	11H	1.051	11H	1.06
18H	1.244	19H	1.027	16H	1.107	16H	1.256
17H	1.339	16H	1.119	18H	1.286	19H	1.259
15H	1.373	17H	1.511	15H	1.338	20H	1.329
12H	1.531	10H	1.52	12H	1.347	15H	1.472
14H	1.617	11H	1.53	14H	1.352	18H	1.579
11H	1.65	14H	1.64	10H	1.379	17H	1.61
10H	1.69	12H	1.719	17H	1.452	10H	1.687
13H	2	13H	2.635	13H	1.637	12H	1.854
46H	3.21	46H	3.196	38H	3.404	31H	2.804
52H	3.449	45H	3.545	32H	3.43	32H	3.973
45H	4.308	52H	3.571	31H	3.986	38H	4.606
61H	5.46	59H	5.359	45H	6.192	65H	6.559
59H	6.121	59H	5.982	47H	6.469	47H	6.656
34H	6.558	37H	6.33	66H	6.74	45H	6.661
37H	6.753	9H	6.675	64H	6.787	36H	6.664
49H	6.763	34H	6.769	65H	6.844	63H	6.671
33H	6.851	51H	6.852	35H	6.801	50H	6.695
51H	6.86	47H	6.894	63H	6.928	64H	6.791
36H	6.9	65H	6.945	36H	6.97	35H	6.812
50H	6.944	36H	6.968	37H	6.999	66H	6.983
35H	6.956	35H	7.002	62H	7.052	37H	7.004
47H	6.994	48H	7.037	34H	7.079	62H	7.141
48H	7.006	49H	7.076	33H	7.116	33H	7.158
9H	7.285	33H	7.089	50H	7.435	34H	7.612

65H	7.568	50H	7.113	51H	7.906	9H	7.839
66H	7.687	66H	7.578	46H	7.945	46H	8.142
60H	7.972	60H	8.155	9H	8.087	51H	8.186

Table 7: Hyper NMR Proton Shift Results of Ugi 233

Figure 19: Structure and Labeling of Ugi 234 Open

Figure 20: Structure and Labeling of Ugi 234 Closed

Index (C)	Shift (C)	Index (CMD)	Shift (CMD)	Index (O)	Shift (O)	Index (OMD1)	Shift (OMD1)	Index (OMD2)	Shift (OMD2)
1-19H	0.83	1-19H	0.884	1-19H	0.907	1-11H	0.801	1-15H	0.704
1-20H	1.202	1-20H	1.239	1-20H	1.066	1-16H	1.027	1-20H	0.839
1-16H	1.35	1-16H	1.382	1-16H	1.324	1-12H	1.105	1-18H	0.894
1-18H	1.401	1-18H	1.453	1-18H	1.445	1-19H	1.405	1-14H	1.06
1-17H	1.469	1-17H	1.554	1-15H	1.538	1-17H	1.516	1-16H	1.156
1-15H	1.514	1-15H	1.555	1-14H	1.619	1-15H	1.533	1-19H	1.166
1-12H	1.7	1-12H	1.721	1-17H	1.606	1-18H	1.554	1-11H	1.195
1-14H	1.763	1-14H	1.783	1-11H	1.629	1-20H	1.822	1-17H	1.547
1-11H	1.809	1-11H	1.818	1-12H	1.625	1-10H	1.941	1-12H	1.655
1-10H	1.845	1-10H	1.869	1-10H	1.723	1-14H	2.507	1-10H	1.876
1-13H	2.117	1-13H	2.153	1-13H	1.971	1-33H	2.724	1-13H	2.34
1-46H	3.397	1-46H	3.46	1-33H	3.124	1-13H	3.008	1-32H	3.726
1-52H	3.921	1-52H	3.927	1-39H	3.816	1-39H	3.712	1-33H	3.952
1-45H	4.489	1-45H	4.5	1-32H	4.098	1-32H	3.91	1-39H	4.091
1-34H	6.633	1-34H	6.57	1-67H	6.854	1-55H	6.456	1-55H	6.606
1-37H	6.878	1-69H	6.65	1-36H	6.926	1-38H	6.53	1-67H	6.903
1-69H	6.938	1-37H	6.853	1-55H	6.972	1-36H	6.619	1-69H	6.957

1-49H	6.945	1-33H	6.908	1-38H	7.018	1-37H	6.689	1-65H	7.034
1-33H	6.996	1-49H	6.965	1-66H	7.054	1-67H	6.755	1-38H	7.054
1-68H	7.023	1-68H	6.995	1-37H	7.074	1-66H	6.927	1-34H	7.156
1-51H	7.044	1-36H	7.055	1-65H	7.176	1-54H	7.041	1-68H	7.171
1-36H	7.082	1-51H	7.069	1-68H	7.18	1-65H	7.264	1-66H	7.182
1-50H	7.121	1-50H	7.149	1-34H	7.266	1-68H	7.382	1-36H	7.214
1-35H	7.143	1-35H	7.207	1-35H	7.273	1-35H	7.433	1-37H	7.386
1-47H	7.251	1-47H	7.259	1-54H	7.299	1-69H	7.546	1-35H	7.394
1-48H	7.267	1-48H	7.282	1-56H	7.307	1-34H	7.581	1-54H	7.518
1-67H	7.39	1-67H	7.379	1-69H	7.593	1-56H	8.215	1-56H	7.574
1-9H	7.773	1-9H	7.872	1-9H	8.725	1-9H	9.505	1-9H	8.762

Table 8: Hyper NMR Proton Shift Results of Ugi 234

Index (C)	Description	Shift (C)	Index (O)	Description	Shift (O)
7C	Cyclohexane	28.753	5C	Cyclohexane	28.303
6C	Cyclohexane	28.858	6C	Cyclohexane	28.712
5C	Cyclohexane	30.347	7C	Cyclohexane	28.712
4C	Cyclohexane	35.759	4C	Cyclohexane	34.817
8C	Cyclohexane	36.011	8C	Cyclohexane	35.887
3C	Cyclohexane	45.256	3C	Cyclohexane	48.242
38C	Benzyl CH2	55.74	25C	Benzyl CH2	57.603
22C	sp3 Adjacent to hexafluoro	69.366	22C	sp3 Adjacent to hexafluoro	70.594
30C	Triple Bond	124.934	57C	Triple Bond	121.817
29C	Triple Bond	129.535	62C	Aromatic	130.24
42C	Aromatic	130.415	29C	Aromatic	130.326

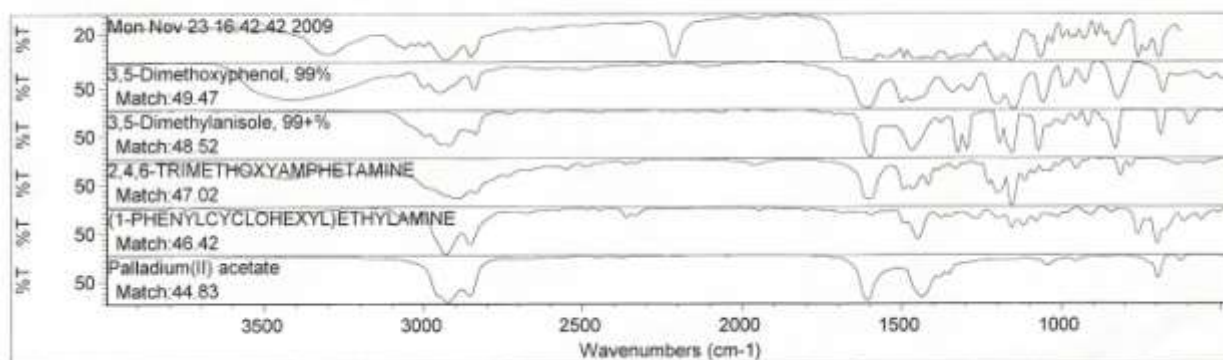
28C	Aromatic	130.627	61C	Aromatic	130.956
43C	Aromatic	131.605	63C	Aromatic	131.003
26C	Aromatic	131.661	30C	Aromatic	131.421
41C	Aromatic	131.729	28C	Aromatic	131.804
23C	Aromatic	132.528	44C	Aromatic	133.106
27C	Aromatic	132.617	42C	Aromatic	133.801
24C	Aromatic	132.656	58C	Triple Bond	135.22
57C	Aromatic	133.048	43C	Aromatic	135.36
55C	Aromatic	133.767	60C	Aromatic	143.359
25C	Aromatic	134.829	59C	Aromatic	144.191
56C	Aromatic	135.448	64C	Aromatic	145.041
40C	Aromatic	146.135	27C	Aromatic	146.511
44C	Aromatic	147.135	31C	Aromatic	147.193
58C	Aromatic	150.753	41C	Aromatic	150.216
54C	Aromatic	151.042	45C	Aromatic	151.041
39C	Aromatic	151.515	26C	Aromatic	151.452
60C	sp ³ Adjacent to hexafluoro	153.532	47C	sp ³ Adjacent to hexafluoro	153.563
59C	sp ³ Adjacent to hexafluoro	153.778	46C	sp ³ Adjacent to hexafluoro	153.855
53C	Aromatic C near hexafluoro	158.583	40C	Aromatic C near hexafluoro	158.017
31C	Carbonyl	184.818	23C	Carbonyl	185.379
1C	Carbonyl	196.548	1C	Carbonyl	196.98

Table 9: Hyper NMR C¹³ Shift Results of Ugi 234

Signal (cm ⁻¹) of Ugi 173G	Peak Identity	Signal (cm ⁻¹) of Ugi 233	Peak Identity	Signal (cm ⁻¹) of Ugi 234	Peak Identity
1600	carbonyl	1600-1700	carbonyl	1630	carbonyl
1632	carbonyl				
2222	triple bond C	2222	triple bond C	2222	triple bond C
3274	amide	3300	amide	3250	amide

Table 10: FT-IR Results of Ugi 233, Ugi 234, and Ugi 173G

Search results for: Mon Nov 23 16:42:42 2009
Date: Mon Nov 23 16:46:04 2009
Search algorithm: Correlation
Regions searched: 3495.26-455.13



Search results list of matches

	Index	Match	Compound Name	Library Name
1	317	49.47	3,5-Dimethoxyphenol, 99%	Nicolet Condensed Phase Academic Sampler
2	972	48.52	3,5-Dimethylanisole, 99+%	Nicolet Condensed Phase Academic Sampler
3	28	47.02	2,4,6-TRIMETHOXYAMPHETAMINE	Georgia State Crime Lab Sample Library
4	196	46.42	(1-PHENYLCYCLOHEXYL)ETHYLAMINE	Georgia State Crime Lab Sample Library
5	890	44.83	Palladium(II) acetate	Nicolet Condensed Phase Academic Sampler
6	90	44.77	3,5-DIMETHYLPHENOL, 99+%	Aldrich Vapor Phase Sample Library
7	67	44.74	PHENYL SULFONE, 97%	Aldrich Condensed Phase Sample Library
8	542	44.37	m-Anilidine, 97%	Nicolet Condensed Phase Academic Sampler
9	145	43.05	Bibenzyl	Nicolet Condensed Phase Academic Sampler
10	481	42.18	3,5-Dimethoxybenzaldehyde, 98%	Nicolet Condensed Phase Academic Sampler

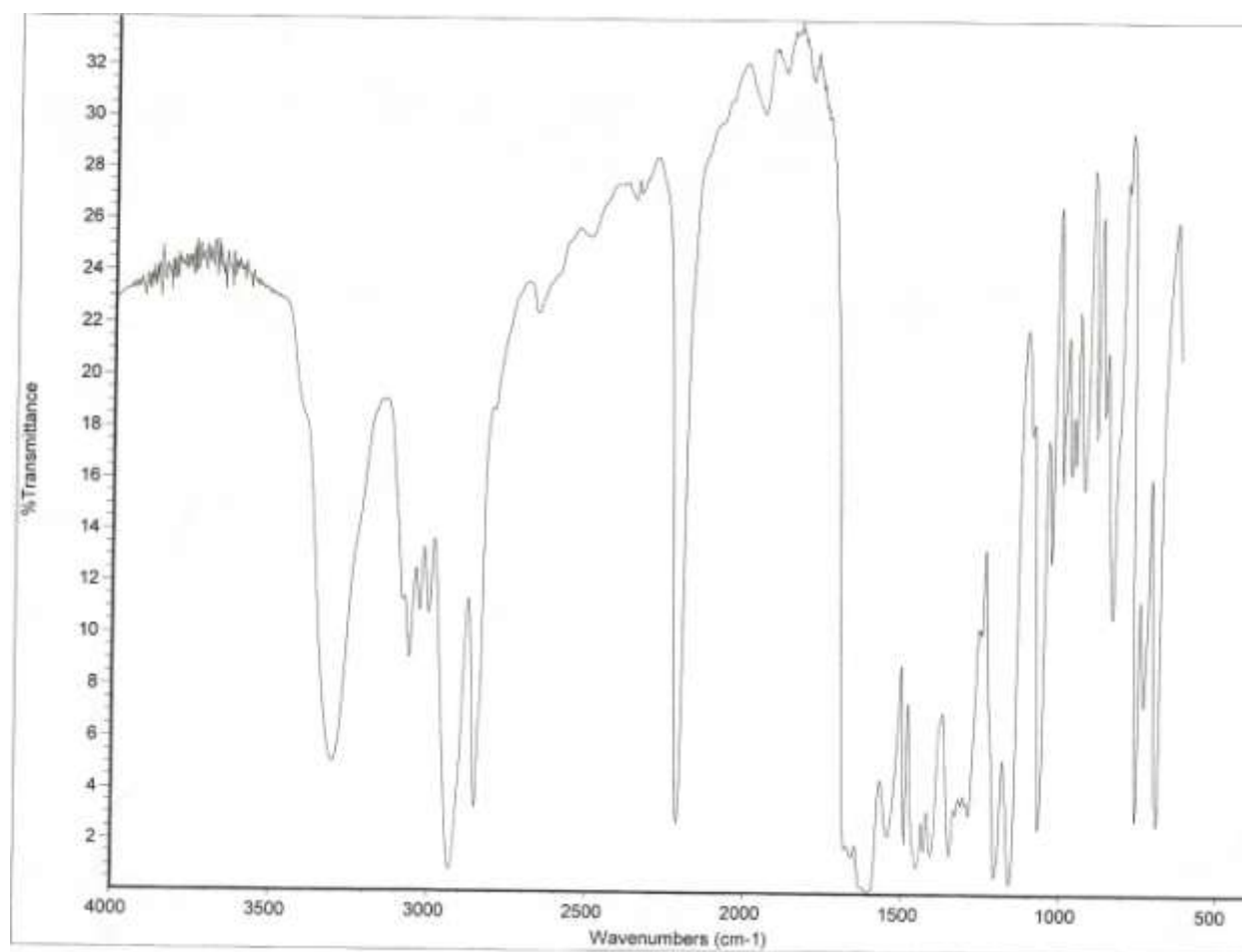
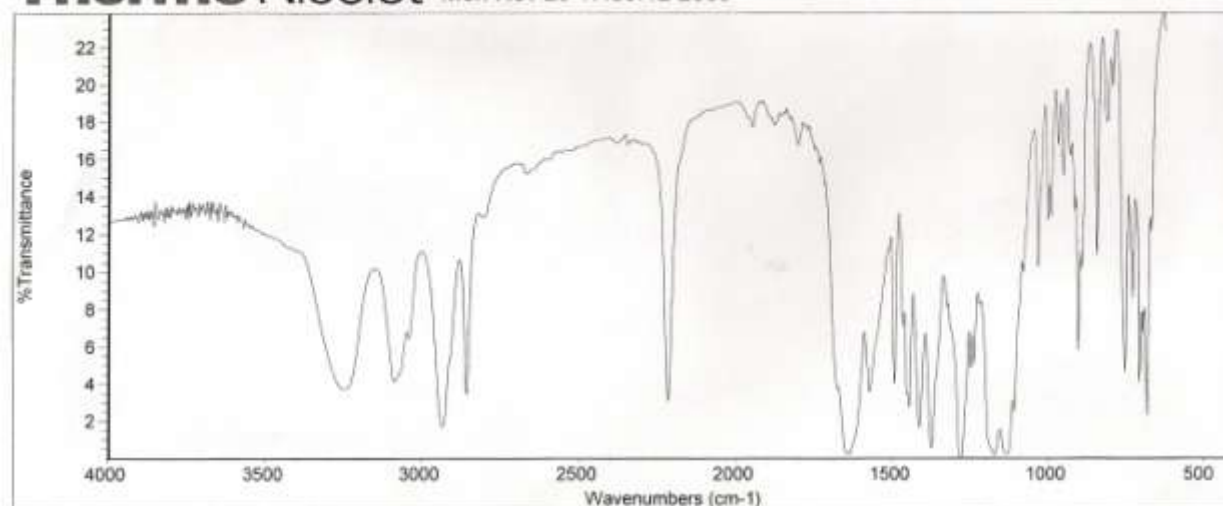


Figure 21: FT-IR with correlation of Ugi 233



Number of sample scans: 32
 Number of background scans: 32
 Resolution: 4.000
 Sample gain: 8.0
 Mirror velocity: 0.6329
 Aperture: 100.00

Spectrum: Mon Nov 23 17:35:12 2009
 Region: 3495.26-455.13
 Search type: Correlation

Index	Match	Compound name
481	37.76	3,5-Dimethoxybenzaldehyde, 98%
697	35.42	Procaine, 99+%
605	33.94	a,a,a-Trifluoro-m-toluic acid, 99%
447	33.53	4'-Aminoacetophenone, 99%
995	33.18	Nylon 6/6
105	32.78	BENZOCAINE IN KBR
388	32.75	1,3-Cyclohexanedione, 97%
66	32.54	VITAMIN K (MENADIOLONE) IN KBR
97	32.36	NYLON 6/10
625	31.53	3-Chloro-2-nitrobenzoic acid, 99%

Library
 Nicolet Condensed Phase Academic Sampler
 Nicolet Condensed Phase Academic Sampler
 Nicolet Condensed Phase Academic Sampler
 Nicolet Condensed Phase Academic Sampler
 Nicolet Condensed Phase Academic Sampler
 Georgia State Crime Lab Sample Library
 Nicolet Condensed Phase Academic Sampler
 Georgia State Crime Lab Sample Library
 Aldrich Condensed Phase Sample Library
 Nicolet Condensed Phase Academic Sampler

Figure 22: FT-IR with correlation of Ugi 234

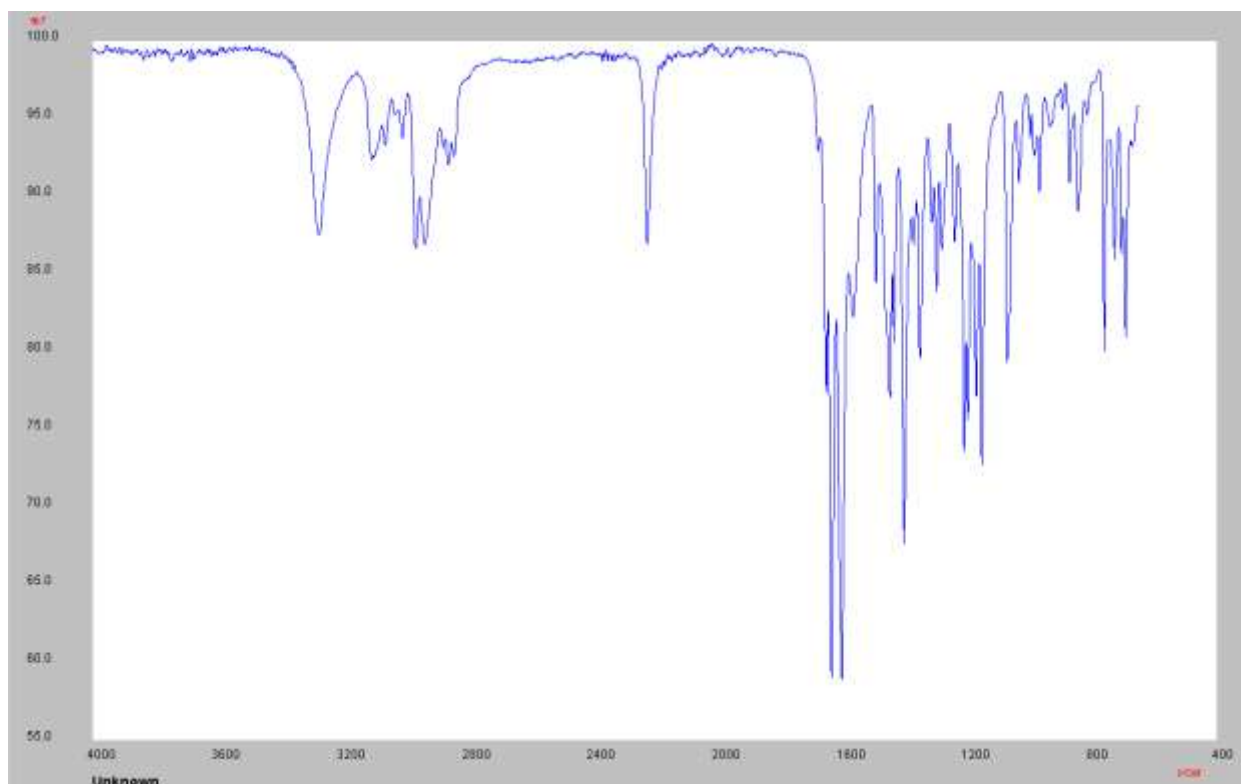


Figure 23: FT-IR of Ugi 173G

Results and Conclusions

The identity of the Ugi products has been confirmed by the spectra in this experiment. The ¹H NMR showed that both Ugi 233 and 234 had very similar, yet indiscernible signals from 0-3 ppm. This corresponds to the cyclohexane ring predicted in the compounds (Figures 1-3, Table 1). The ¹H NMR of Ugi 175G was very different because it has a tert-butyl group instead of the cyclohexane ring. Otherwise, Ugi 175G is identical to the predicted structure of Ugi 233. This is evidenced by a comparison of the close up on the aromatic region (~7 ppm). The two regions are the same except for the resolution between signals due to the higher magnetic field NMR (500 MHz) being run on Ugi 175G compared to the 300 MHz NMR, which was used in this experiment.

The carbon 13 NMR confirmed the results of the proton NMR with greater precision due to the wider range of carbon 13 peaks (Table 2, Figures 4-6). From about 24 to 50 ppm, the peaks are similar between Ugi 233 and Ugi 234. The four carbon peaks correspond to the cyclohexane due to identical shielding of C5 and C7 as well as C4 and C8 (Numbering designations from Figures 16-18). The C6 would be expected to be the least shielded, then the C5 and C7 peak, then the C4 and C8 peak, and the most shielding being the C3 peak in Ugi 233 and Ugi 234. The next carbon signal (51-52 ppm) is approximately the same throughout the three Ugi Products. At this chemical shift, it fits best with the benzylic carbon, C24 in Ugi 233 and C38 in Ugi 234. The next two peaks (~55 and 62-63 ppm) of Ugi 173G has corresponding peaks in Ugi 233 at about the same chemical shift, indicating possible correlation with the methoxy group. The triplet at 77 ppm corresponds to the solvent, chloroform-d. Then the next two peaks (81-82 ppm and 91-92 ppm) can be found in all three Ugi products, possibly correlating to the triple bonded carbons. The rest of the peaks are mostly aromatic carbons found in the many aromatic rings, except for a couple carbonyls found far downfield. The close-ups show nearly identical cyclohexane regions between Ugi 233 and Ugi 234 and nearly identical aromatic regions between Ugi 233 and Ugi 175G.

Extra peaks appeared in DEPT135 that do not show protons on the chshf. There have been problems with the DEPT135 this semester. A quick fix was attempted by widening the peaks. However, this does not seem to have worked. The DEPT90 spectrum contains even more extra peaks and contains all of the peaks found in the DEPT135, which was not expected. This could be explained since the DEPT90 has had even more problems than the DEPT135 and a fix has not been attempted. Therefore, the DEPT will not be used in this experiment. The chshf spectrum correlates the proton and carbon 13 spectra in Ugi 233 and 234 (Tables 3 and 4). This not only

shows which carbons have protons, but also gives more information about the identity of the atoms. For example, the carbon 13 NMR shows clear peaks for the cyclohexane, but the proton NMR is indiscernible for cyclohexane. By the chshf, it can be seen that the blur of signals in the proton NMR do correspond to the clear peaks in the carbon 13 NMR. This can be implemented to tie the two spectra together to glean more information about the identity of the compounds.

The COSY shows intramolecular proton interactions on adjacent carbons (Tables 5 and 6). This aided in the determination of the identity of the protons in Table 1. Interactions are shown within the cyclohexane ring and between some of the aromatic protons. In Useful Chemistry Experiment 173G, Khalid Mirza hypothesized that Ugi 173G showed sets of major and minor peaks in a 3:1 ratio due to the existence of rotamers around the triple bonded group. Both Ugi 233 and 234 in this experiment seem to have a triple bonded group. Therefore, the HyperChem software was utilized to draw out the open and closed rotamers, and then use HyperNMR to predict the chemical shifts. These chemical shifts were somewhat different. However, the prediction is not very accurate in practice due to solvent and temperature effects. The NOESY demonstrates intramolecular proton interactions through space, and does not always correspond to the adjacent carbons. This information is used to determine the structural conformation. The interaction in Ugi 233 between the methoxy protons at 3.65 ppm and an aromatic proton at 6.58 ppm, either 35H indicating the closed conformation or 47H indicating the open conformation. The aromatic proton also shows interactions with the proton singlet at 5.94 ppm, which corresponds to the carbon at 62 ppm. This carbon is most likely 22C. Therefore, the proton singlet is most likely 52H. In other words, either 47H or 35H is interacting with either 65H or 66H while also interacting with 52H. By the way, labeling refers to that of Ugi 233 closed. In Figure 17 (Open), 33H (same as 47H in closed) is spatially close to 38H (same as 52H in closed)

and 50H (same as 65H in closed). On the other hand, Figure 18 (Closed) shows neither 47H or 35H being spatially close to the methoxy protons. Therefore, it appears that the open position of Ugi 233 is more favorable than the closed. Ugi 234 shows interactions between the proton at 7.654 ppm and the proton at 6.198 ppm. The proton at 6.198 ppm also shows interactions with a proton at 6.327 ppm. The chshf shows that the carbon at 60.8602 ppm is attached to the proton at 6.198 ppm and the proton at 6.327 ppm, as well as that the carbon at 129.8644 ppm is attached to the proton at 7.654 ppm. The proton at 7.654 ppm is one of three aromatic proton singlets far downfield that most likely corresponds to the three protons on the 3,5-bis trifluoromethyl aromatic ring. This would be 56H in the open conformation and 69H in the closed conformation due to the shielding the nearby carbonyl. The carbon at 60.8602 ppm seems to be 22C, and the carbon at 129.8644 ppm is the corresponding aromatic carbons (45C in open or 58C in closed). The proton attached to 22C at 6.198 ppm would then be 52H if closed or 39H if open. The two proton chemical shifts attached to 22C correspond to the same the proton. However, this information is not quite enough to determine the conformation. Another possibility to be considered later on would be to determine which enantiomer is most prevalent around the chiral 22C.

The FT-IR of Ugi 233 and 234 is almost identical to that of Ugi 178G, except for the maxing out of the fingerprint region as a result of too much sample in the KBr disc in Ugi 233 and 234. The spectrum indicates carbonyls at around 1630 cm^{-1} , triple bonded carbon stretch at 2222 cm^{-1} , and the amide at about 3270 cm^{-1} . Overall, the results indicate that Ugi 233 is

COc1cc(cc(c1)OC)C(C(=O)NC2CCCCC2)N(Cc3ccccc3)C(=O)C#Cc4ccccc4 in SMILES notation, and Ugi 234 is

c1ccc(cc1)CN(C(c2cc(cc(c2)C(F)(F)F)C(F)(F)F)C(=O)NC3CCCCC3)C(=O)C#Cc4ccccc4 in
SMILES notation.